Interlaboratory Study 2002-1 Toxicity Characteristic Leachate Procedure (TCLP)

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Ontario

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(TCLP)

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1. INTRODUCTION

The leachate procedure specified under Ontario Regulation 347, (formerly Reg 309)[1] is an adaptation of the US-EPA Method 1311, Toxic Characteristic Leachate Procedure (TCLP)[2]. The leachate procedure is used to characterize solid waste material based on the ability of contaminants to partition, or leach, into a simulated landfill solution. The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiple phase wastes under acidic conditions.

A leachate toxic waste, is defined as waste that produces a leachate, using a specified procedure, that contains any of the contaminants at a concentration equal to, or greater than that specified in Schedule 4 of Regulation 347.

There are three possible scenarios for a waste sample received at a laboratory for analysis, that are listed below:

1. The waste (original sample) is less than 0.5% (< 0.5%) solid, in which case the solid is disregarded and the liquid (filtrate) is deemed the TCLP extract. Proceed to analysis.

2. The waste (original sample) is more than or equal to 0.5% (≥ 0.5%) solid, in which case the solid is leached. The prepared leachate is filtered and may be combined with the liquid (filtrate) to produce the TCLP extract. Proceed to analysis.

3. The waste is 100 % solid, no liquid is expressed when pressure filtered at 50 psi, in which case the solid is leached, the leachate is filtered and the filtered leachate is deemed the TCLP extract. Proceed to analysis.

If screening analysis indicates that the bulk material contains less than 20 times the acceptable leachate level, as specified in Schedule 4, then no further analysis is required. If the waste contains both solid and liquid, and the liquid contains equal to or greater than the acceptable leachate level, as specified in Schedule 4, then no further analysis is required.

1.1 Principal of TCLP Method [3]

A 100 gram sample of pressure filtered "de-watered" solid waste is leached for 18 ± 2 hours with 2.0 liters of leaching fluid. If a smaller sample size is used, then the amount of leaching fluid must be adjusted in order to maintain a 20:1 ratio of leaching fluid to solid waste. There are two options for the leaching solution. After preparing the "de-watered" waste, a small aliquot of waste is made into a slurry to test the pH. If the pH is ≤5.0, use extraction fluid #1 (buffer - dilute glacial acetic acid/sodium hydroxide mixture). If the pH is ≥5.0, use extraction fluid #2 (acid - dilute glacial acetic acid). See Appendix 5 for a flowchart of this procedure. The prepared leachate from this method is distributed to the required analytical methods to determine the concentration of the Schedule 4 contaminants.

Several 100 gram sample aliquots may need to be leached in order to produce sufficient leachate to accommodate all required methods.

1.2 Interlaboratory Study Goals

The Quality Management Unit (QMU) of the Laboratory Services Branch (LSB), Ontario Ministry of the Environment (MOE), initiated this TCLP interlaboratory study (ILS) to assess the between-laboratory performance of Canadian laboratories who may be providing data for Regulation 347. This performance assessment is intended to provide an overview of the current application of the TCLP method and identify areas for improvement. The target parameters for this study were trace metals, as listed in Table 1.

Table 1 - Study Target Analytes		
*Aluminum (Al)	Cobalt (Co)	Molybdenum (Mo)
Barium (Ba)	Copper (Cu)	Nickel (Ni)
Beryllium (Be)	Iron (Fe)	Strontium (Sr)
*Cadmium (Cd)	*Lead (Pb)	Vanadium (V)
Calcium (Ca)	Magnesium (Mg)	Zinc (Zn)
*Chromium (Cr)	Manganese (Mn)	

* Regulated parameters

The study design of more target parameters than just the regulated elements, was intended to provide additional information for the participants and MOE-LSB. Trace metal analysis is most commonly done using ICP, a multi-element technique, so that the reporting of additional parameters would not increase the cost to the participants when analyzing the study samples. Data on other leachable metals that is provided by the study participants may be beneficial for MOE regulators if modifications to Regulation 347 is required.

In addition, the QMU hoped that sufficient data from the analysis of the two solid waste materials used in this study would be generated, so that the two wastes could be characterized as reference materials for this procedure.

A letter of invitation was sent to members of the Canadian Association of Environmental Analytical Laboratories (CAEAL) in the fall of 2001. Laboratories who agreed to participate were provided with a copy of LSB Method E9002 - The Preparation of Leachates Using the Toxicity Characteristic Leaching Procedure (TCLP)[3]. Each laboratory was assigned a confidential laboratory identification code. A list of participating laboratories is provided in Appendix 4. A copy of all correspondence is provided in Appendix 5.

2. STUDY DESIGN

The study was designed to assess the trace metals listed in Table 1. Each participating laboratory would receive two ampouled, trace metal standards, to assess calibration comparability. Each participant would receive two "leachates", to assess instrumental, including digestion, analytical comparability. Each participant would also receive two solid wastes to be processed through the complete TCLP and analyzed for the target trace metals, to assess the full between-laboratory performance for the TCLP.

2.1 Preparation of the Standards

The two standards were prepared in bulk (6 L each) by the LSB QMU, by appropriately diluting two commercial standards and one in-house standard into acidified, deionized water. The bulk standards were throughly mixed and were dispensed into ampoules, sealed, labeled and stored at room temperature.

2.2 Leachate Sample Preparation

Leachate Sample 1

Twenty (20) portions of 50 g each of garden soil were weighed out and then loaded into 20 different 1250 mL HDPE (high density polyethylene) bottles. One liter of fluid #2(acid) [3] was added to each bottle. The bottles were capped and then loaded into 2 large rotary tumblers and tumbled for approximately eighteen hours (18) at thirty (30) revolution per minute (rpm). Each leachate sample was then filtered through an acid washed glass fiber filter (GFF, 0.7 microns). The filtrate was composited in a 20 liter plastic carboy bottle.

Leachate Sample 2

Twenty (20) portions of 50 g each of garden soil were weighed out and then loaded into 20 different 1250 mL HDPE bottles. One liter of fluid #1(buffer) [3] was added to each bottle. The bottles were capped and then loaded into 2 large rotary tumblers and tumbled for approximately eighteen hours (18) at thirty (30) revolution per minute (rpm). Each leachate sample was then filtered through an acid washed glass fiber filter (GFF, 0.7 microns). The filtrate was composited in a 20 liter plastic carboy bottle.

The two leachate solutions were spiked with trace element standard solutions containing, aluminum, cadmium, chromium and lead, such that the final solution of Leachate 1 and Leachate 2 contained the above, regulated elements, at approximately 80% and 120% respectively of the regulatory limit. The samples were preserved to pH <2.0 using nitric acid. The bulk samples were thoroughly mixed before dispensing to individual 250 PET sample bottles.

2.3 Solid Waste Sample Preparation

The Solid Waste samples were prepared by mixing two industrial wastes in varying amounts. Geoscience Laboratories of Sudbury, Ontario, certified to ISO 9001 standards, were contracted to perform the physical processing. The stages of processing included air drying, sieving, milling, mixing, dispensing into bottles, labeling, etc.

The materials were assessed for homogeneity by randomly selecting 12 bottles of each material and analyzing in triplicate for several trace metals. ANOVA was employed to demonstrate that the material were homogeneous with respect to these analytes[4].

2.4 Sample Distribution

Sample sets, consisting of the two ampouled Standards, two Leachates of approximately 250 mL in PET bottles, and two Solid Wastes of 110 g each, were sent by courier to the participants on February 27, 2002. An instruction sheet and report form were included with the samples, as well as a methodology questionnaire. Examples are included in Appendix 6. Electronic versions of the report form and methodology questionnaire were provided on diskette to all of the participants.

3. STUDY RESULTS

Twenty nine laboratories reported results. Most laboratories reported electronically via e-mail. One laboratory reported two sets of results employing two different instrumental techniques, and thus counted as two separate participants to make a total of thirty sets. All except one reported results for all six samples. Laboratory 2010 did not report results for the Standards.

Individual results, as they were received, were transferred into an electronic spreadsheet. Preliminary tables of results were forwarded to participants on July 15, 2002, to verify the accuracy of our transcription. Six labs responded with requests for change.

4. DISCUSSION

4.1 Standards

The results of the Standards were analyzed using the LSB KS procedure[5], which is based on Youden-type plots. In this procedure, the results (as percentage of the appropriate median) of one standard is plotted versus the second standard for each parameter. In this X-Y plot, a vertical and horizontal line at the median value are drawn, dividing the graph into four quadrants. As well, a 45° line is also drawn between the origin and through the intersection of the median values. Laboratories whose results fall on the 45° line demonstrate good within-laboratory precision. The spread of results along this line in the upper right and lower left

quadrants indicates the between-laboratory bias (high or low). Results in the upper left and lower right quadrants are considered erratic (one example has been provided in Appendix 2, Figure 1).

A circle, centered on the intersection of the ILS medians and with a radius of the reference uncertainty value (two times study relative standard deviation, SD), has also been included in each graph. Participants whose results are inside the circle, have demonstrated satisfactory performance. Laboratories whose results are outside the circle but within a radius of three times SD are flagged as "Warning". Those laboratories whose results are outside the 3 SD circle (not drawn) are flagged as high bias, low bias or erratic, based on their position on the graph. Laboratories that reported results for only one sample or reported as not detected were flagged as "other" in Table 4.

Most laboratories demonstrated a good degree of precision, but biases among themselves were clearly evident. The study medians for the all the analytes of both samples were within three standard deviations of the design value of the standards. Tables 2 and Table 3 provide the statistical summary for the two standards.

Table 2 - Standard 1 (Sample 02-1-1)
Statistical Summary

Analyte	Design mg/L (D)	Study Median mg/L (M)	Std. Deviation mg/L (SD)	CV (SD/M)*100	Number of Labs
Aluminum	0.50	0.61	0.04	6.56%	22
Barium	0.10	0.10	0.0054	5.40%	25
Beryllium	0.10	0.10	0.0052	5.25%	26
Cadmium	0.10	0.10	0.006	6.00%	28
Calcium	1.25	1.30	0.086	6.62%	16
Chromium	0.10	0.10	0.0057	5.70%	27
Cobalt	0.10	0.10	0.0047	4.70%	28
Copper	0.08	0.08	0.0052	6.84%	23
Iron	1.00	1.00	0.067	6.70%	27
Lead	0.50	0.50	0.026	5.20%	26
Magnesium	0.50	0.49	0.026	5.33%	23
Manganese	0.30	0.30	0.01	4.0%	23
Molybdenum	0.40	0.39	0.04	9.9%	27
Nickel	0.40	0.40	0.01	2.5%	22
Strontium	0.40	0.39	0.01	3.3%	24
Titanium	0.40	0.40	0.02	5.7%	18
Vanadium	0.40	0.40	0.02	3.8%	24
Zinc	0.50	0.51	0.02	4.6%	25

Table 3 - Standard 2 (Sample 02-1-2)
Statistical Summary

Analyte	Design mg/L (D)	Study Median mg/L (M)	Std. Deviation mg/L (SD)	CV (SD/M)*100	Number of Labs
Aluminum	2.00	2.08	0.10	4.8%	26
Barium	0.40	0.39	0.02	4.6%	25
Beryllium	0.40	0.40	0.02	4.5%	27
Cadmium	0.40	0.40	0.02	5.5%	27

Table 3 - Standard 2 (Sample 02-1-2)
Statistical Summary

Analyte	Design mg/L (D)	Study Median mg/L (M)	Std. Deviation mg/L (SD)	CV (SD/M)*100	Number of Labs
Calcium	3.75	3.80	0.24	6.4%	24
Chromium	0.40	0.40	0.01	3.5%	27
Cobalt	0.40	0.39	0.01	3.6%	27
Copper	0.30	0.30	0.01	4.7%	28
Iron	4.00	3.96	0.17	4.3%	26
Lead	2.00	2.00	0.10	4.8%	27
Magnesium	1.50	1.46	0.07	4.3%	25
Manganese	0.08	0.08	0.004	5.33%	25
Molybdenum	0.10	0.10	0.008	8.00%	24
Nickel	0.10	0.10	0.0024	2.40%	19
Strontium	0.10	0.10	0.0048	4.80%	25
Titanium	0.10	0.10	0.0048	4.80%	18
Vanadium	0.10	0.10	0.0051	5.10%	26
Zinc	0.13	0.13	0.0068	5.23%	22

Table 4 provides a summary of performance of individual laboratories for the standards. Performance is considered satisfactory if their score is 12 or greater.

Table 4 - Standards (Samples 02-1-1 & 02-1-2)
Performance Summary

ID Code	Satisfactory	Warning	Score (Satisfactory + Warning)	High Bias	Low Bias	Erratic	Other
2101	10	0	10	4	2	2	0
2102	12	0	12	3	0	2	1
2103	7	0	7	1	4	3	3
2104	14	0	14	2	1	1	0
2105	15	0	15	0	1	2	0
2106	15	1	16	0	1	0	1
2107	7	2	9	0	4	0	5
2108		0	0	0	15	0	3
2109	17	0	17	0	0	0	1
2111	11	2	13	0	1	3	1
2112	13	0	13	1	0	1	3
2113	1	3	4	14	0	0	0
2116	9	2	11	6	0	1	0
2117	16	0	16	1	0	1	0
2118	13	0	13	0	2	2	1
2119	14	1	15	1	1	1	0
2120		1	1	10	0	7	0
2121	13	1	14	3	0	0	1
2122	7	0	7	0	1	10	0
2123	17	0	17	0	1	0	0

Table 4 - Standards (Samples 02-1-1 & 02-1-2)
Performance Summary

ID Code	Satisfactory	Warning	Score (Satisfactory + Warning)	High Bias	Low Bias	Erratic	Other
2124	12	0	12	4	0	1	1
2125	12	2	14	1	3	0	0
2126	14	1	15	2	1	0	0
2128	15	0	15	1	1	1	0
2130	12	1	13	2	1	2	0
2131	13	0	13	0	3	1	1
2132	4	1	5	3	9	1	0
2133-ICP	6	0	6	1	10	1	0
2133-ICP/MS	6	0	6	0	8	3	1

4.2 Leachates

The results of the leachates were also analyzed using the LSB KS procedure. Most laboratories demonstrated a good degree of precision, but again, inter-laboratory bias was evident. Table 5 provides the statistical summary of the samples. Table 6 provides a summary of performance of individual laboratories.

Table 5 - Leachates (Samples 02-1-3 & 02-1-4)
Statistical Summary

Analyte	Leachate 1				Leachate 2			
	Study Median mg/L (M)	Std. Deviation mg/L (SD)	CV (SD/M)*100	Number of Labs	Study Median mg/L (M)	Std. Deviation mg/L (SD)	CV (SD/M)*100	Number of Labs
Aluminum	0.139	0.082	57.5%	24	1.35	0.147	10.9%	27
Barium	0.48	0.02	4.2%	25	0.232	0.022	9.5%	26
Cadmium	0.4185	0.029	6.9%	30	0.573	0.056	9.8%	30
Calcium	227.5	11.46	5.0%	28	186.5	7.54	4.0%	26
Chromium	4.215	0.26	6.2%	30	5.8	0.435	7.5%	29
Copper	0.1	0.01	10.0%	27	0.092	0.014	15.2%	28
Iron	0.2675	0.03	11.2%	24	0.082	0.024	29.3%	18
Lead	4.17	0.191	4.6%	28	5.74	0.337	5.9%	26
Magnesium	22.5	1.13	5.0%	28	14	0.756	5.4%	23
Manganese	3.45	0.212	6.1%	28	0.76	0.08	10.5%	29
Strontium	0.45	0.035	7.8%	29	0.3815	0.035	9.2%	28
Zinc	0.17	0.009	5.3%	20	0.437	0.04	9.2%	25

Based on the KS procedure, a passing score is considered 8 or better (out of a maximum of 12). The majority of the laboratories obtained a score of 8 or higher out of the maximum possible of 12 (Table 6). The KS procedure is, by design, based on within-laboratory repeatability. When the between-laboratory reproducibility (bias) is considerably higher than the within-laboratory repeatability, a greater percentage of failures is expected. The KS procedure allows a tolerance factor of 1.5 for the ratio of within-laboratory repeatability to between-laboratory reproducibility, but this factor of 1.5 is not sufficient to compensate for the between laboratory bias in this study.

Table 6 - Leachates (Samples 02-1-3 & 02-1-4) Performance Summary

ID Code	Satisfactory	Warning	Score (Satisfactory+ Warning)	High Bias	Low Bias	Erratic	Other
2101	2	4	6	3	2	1	0
2102	10	1	11	0	0	0	1
2103	3	4	7	1	2	1	1
2104	8	0	8	2	0	2	0
2105	9	2	11	1	0	0	0
2106	7	1	8	1	0	3	0
2107	5	1	6	2	1	0	3
2108	4	0	4	0	6	2	0
2109	8	0	8	1	1	2	0
2110	5	3	8	1	1	2	0
2111	6	0	6	1	4	1	0
2112	4	2	6	0	1	1	4
2113	5	3	8	2	0	0	2
2116	8	2	10	1	0	1	0
2117	7	2	9	1	0	1	1
2118	1	2	3	0	6	3	0
2119	9	1	10	0	2	0	0
2120	5	3	8	3	0	1	0
2121	7	3	10	0	2	0	0
2122	9	1	10	0	0	0	2
2123	6	3	9	1	0	1	1
2124	8	1	9	2	1	0	0
2125	4	2	6	1	2	3	0
2126	2	3	5	1	4	2	0
2128	1	3	4	2	1	5	0
2130	8	1	9	1	1	1	0
2131	8	3	11	0	1	0	0
2132	5	0	5	0	6	1	0
2133-ICP	8	2	10	0	0	2	0
2133-ICP/MS	6	0	6	0	5	0	1

The laboratories were also evaluated by a broader criteria (i.e. between-laboratory standard deviation SD_{bet}). The performance of any laboratory whose result for an analyte deviates from the corresponding median by less than or equal to three SD_{bet}, is considered successful and flagged as 'satisfactory'. If the deviation was greater than three SD_{bet}, the laboratory was flagged as 'not satisfactory'. This evaluation was carried out for each analyte of both samples and the summary is provided in Table 7.

Table 7 - Leachates (Samples 02-1-3 & 02-1-4)
Summary of Flags

ID Code	LEACHATE 2			LEACHATE 1		
	Satisfactory	Not Satisfactory	Below Lab's Detection limit	Satisfactory	Not Satisfactory	Below Lab's Detection limit
2101	9	3		7	5	
2102	11		1	12		
2103	10	1	1	9	2	1
2104	11	1		12		
2105	12			12		
2106	10	2		10	2	
2107	7	2	3	9	2	1
2108	11	1		8	4	
2109	11	1		11	1	
2110	11	1		11	1	
2111	11	1		12		
2112	9		3	11		1
2113	10		2	12		
2116	12			12		
2117	11		1	11	1	
2118	9	3		10	2	
2119	12			12		
2120	12			11	1	
2121	12			12		
2122	9		3	9		3
2123	10	1	1	12		
2124	12			11	1	
2125	11	1		10	2	
2126	11	1		12		
2128	11	1		10	2	
2130	12			11	1	
2131	12			12		
2132	10	2		12		
2133-ICP	11	1		11	1	
2133-ICP/MS	9	2	1	9	2	1

4.3 Solid Wastes

Only analytes for which the majority of laboratories reported positive results (i.e. greater than laboratory detection limit) were evaluated. 12 analytes met this criteria.

The KS Procedure was applied to the two Waste materials. If no systematic errors are present, one would expect results to be equally distributed among the four quadrants as each of the following combinations will have the same probability of occurrence:

High in Waste 1, High in Waste 2 (upper right quadrant)
High in Waste 1, Low in Waste 2 (upper left quadrant)
Low in Waste 1, High in Waste 2 (lower right quadrant)
Low in Waste 2, Low in Waste 2 (Lower left quadrant)

When the majority of the results are in the lower left quadrant or in the upper right quadrant, as in these cases, it is reasonable to conclude that systematic errors are the dominant contributor to the overall variability of the results. The distribution of the majority of results for all the parameters in the lower left and upper right quadrant also confirms the that the waste materials are homogeneous with respect to these analytes.

The fact that the results of the majority of the laboratories lie close to the 45 degree line, (Appendix 2, Figures A2-3, A2-5, A2-8, A2-10, and A2-13) indicate that most laboratories are capable of precise analysis, for certain elements.

Table 8 - Solid Wastes (Samples 02-1-5 & 02-1-6)
Statistical Summary

	WASTE 1				WASTE 2			
Analyte	**Mean mg/L**	**STD mg/L**	**CV**	**n**	**Mean mg/L**	**STD mg/L**	**CV**	**n**
Ba	0.314	0.0825	26.3%	23	0.389	0.041	10.5%	16
Ca	1870.13	229.74	12.3%	24	1741.46	115.71	6.6%	20
Cd	0.23	0.185	80.4%	20	0.781	0.402	51.5%	20
Cr	0.181	0.064	35.4%	24	0.125	0.041	32.8%	24
Cu	0.03	0.022	73.3%	16	0.027	0.017	63.0%	18
Fe	0.121	0.134	110.7%	13	0.117	0.147	125.6%	10
Mg	90.025	13.9	15.4%	22	112.059	16.62	14.8%	22
Mn	0.1497	0.123	82.2%	17	1.26	0.753	59.8%	20
Mo	0.14	0.085	60.7%	22	0.067	0.036	53.7%	21
Pb	0.157	0.119	75.8%	19	0.449	0.311	69.3%	22
Sr	0.96	0.208	21.7%	27	0.958	0.176	18.4%	27
Zn	6.425	7.13	111.0%	23	25.985	23.17	89.2%	24

The 'Box and Whisker' technique, employing a commercially available program 'Quality Analyst'®, was applied to identify outliers. The box and whisker plot consists of a box, whiskers and outliers. A line, drawn at the median, transects the

box. The lower edge of the box is drawn at the first quartile (Q1, the 25th percentile) and the upper edge at the third quartile (Q3, the 75th percentile). The whiskers, the lines extending from the top and bottom of the box, join the highest and lowest values that occur in the regions 1.5 'interquartile range' (Q3 - Q1) above and below the third and first quartiles. All values that fall outside these extremes, outliers, are identified by stars (see example Appendix 2, Figure A2-3). These extreme values were removed from the data pool and a box and whisker plot was re-drawn with the remaining data. This process was repeated until no further outliers could be identified. The remaining data were designated as the final data set. For each parameter, the mean, standard deviation and coefficient of variation (CV) were calculated on the final data set. The summary of the 'Box and Whisker' evaluation is presented in Table 8. The individual box and whisker plots are not included in this report, but histograms showing the distribution of the final data set for each waste are provided in Appendix 2, Figures A2-4 to A2-15.

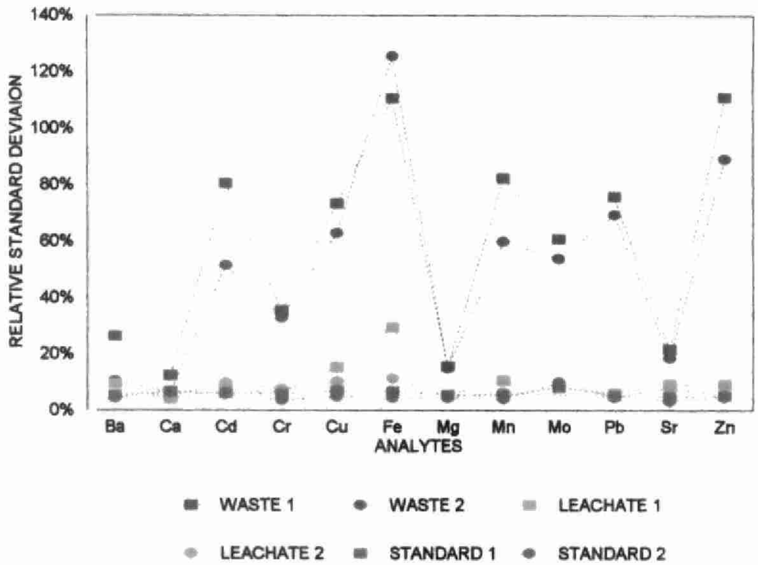
Table 9 presents the number of parameters for which each laboratory is included in the final data set of each waste. For example, the results of laboratory 2126 for all parameters were included in the final data set for both samples and the results of laboratory 2132 were included in only in the final data set of two parameters of both samples.

TABLE 9 - Number of Parameters included in Final Data Set for Solid Wastes

ID Code	Waste 1	Waste 2	ID Code	Waste 1	Waste 2
2101	6	7	2118	10	10
2102	8	10	2119	10	11
2103	9	10	2120	3	4
2104	11	11	2121	8	6
2105	10	10	2122	5	3
2106	10	10	2123	4	4
2107	5	5	2124	9	11
2108	5	4	2125	10	9
2109	4	3	2126	12	12
2110	9	9	2128	11	10
2111	6	4	2130	9	9
2112	9	8	2131	12	11
2113	10	11	2132	2	2
2116	11	12	2133-ICP	12	12
2117	8	6	2133-ICP/MS	11	10

Figure 1 shows the study standard deviations expressed as relative standard deviation(RSD) for the twelve analytes for the six samples of this study. While the RSDs of standards and leachates are comparable, the RSD of the waste

Figure 1 - Between-Laboratory Performance of Study Materials



materials is considerably higher for most analytes. This seems to suggest that the largest contributor to the high variability is the leaching process procedure, and not the instrumental analysis. The fact that the laboratories are demonstrating reasonable precision seems to suggest an inherent difference among laboratories in the leaching process.

Differences in application of the leaching process between the participants is demonstrated in the summary of responses for the preparation of Waste 1 and 2 (Table A3-1, Appendix 3). The methodology questionnaire asked the participants to measure the pH of the Wastes at specific times during the preparation. The first time the pH is measured, is during an initial pre-screening of the sample. The range of "pH Init" for Waste 1 was 10.37 to 12.84, and for Waste 2 was 10.06 to 11.93. The range of pH units is 2.47 and 1.87 for the two samples respectively, and does not appear too wide.

To determine which leaching fluid is required for the sample, a small amount of hydrochloric acid is added to the sample, and heated, if necessary[3]. This is outlined in a flowchart in Appendix 5. The final pH obtained after this step should have been reported as "pH Final". As seen in Table A3-1, the results from the participants vary from 2.64 to 10.66 for Waste 1, and from 0.94 to 11.23 for Waste 2. The participants were also asked to report the pH of the leachate obtained after the 18 hour leaching process ("pH Final Leachate"). The results reported by the participants in Table A3-1 vary from 4.82 to 10.81 for Waste 1, and from 5.2 to 11.01 for Waste 2.

The ranges of the "pH Final" (Waste 1 -8.02, Waste 2 - 10.29) and the "pH Final Leachate" (Waste 1 - 5.99, Waste 2 - 5.81) values reported by the participants suggests differences in the application of the leaching process by the laboratories. Allowing for between-laboratory variability of the pH analysis and the high level of homogeneity of the Waste samples[4], the range of pH values for "pH Final" and for "pH Final Leachate" should be similar to the between-laboratory ranges of

"pH Init", given that the participants are all analyzing the same materials. More detailed questions than those used in this study would be required to determine what the possible sources of variability are between the participants.

The value determined by a laboratory for "pH Final" dictates which extraction fluid is used (Fluid #1 - buffer or Fluid #2 - acid)[3]. The broad range of values reported by the participants for "pH Final" resulted in some of the participants using Fluid #1, though the majority used Fluid #2. This will contribute to the broad range of values for "pH Final Leachate".

The information provided in Tables A3-1 and A3-2 indicate some of the differences in application of the leaching process to the samples. While the method[3] recommends a 50 g sample size, it stresses the importance of maintaining the correct extraction fluid:sample weight ratio. All of the participants maintained the required 20:1 ratio, regardless of the weight of sample used. There is no correlation between sample size and analytical results that were biased high or low.

Similarly, while several participants used a finer filter type (45 micron instead of the recommended 70 micron, Table A3-2), there was no correlation between filter type and analytical results that were biased high or low.

5. CONCLUSION

The between-laboratory performance for the Standards and Leachates showed good comparability. The interlaboratory median for the Standards was within three standard deviations of the design value, indicating that most participants had good between-laboratory agreement. As described above in Figure 1, the between-laboratory performance for the Leachates is similar to the Standards. The variability in the data set for the Wastes and the variability in the pH data for the Wastes analysis, indicates differences between the participants in the application of the TCLP method, but there is no clear correlation with other information provided in the methodology questionnaire. More detailed information on the application of the TCLP method may be required to identify the sources of the differences between the laboratories. The effects of these differences may be amplified in wastes containing 'leachable' metals at low levels. Further intercomparison exercises may be required to resolve these differences.

The results from this interlaboratory study did not provide sufficient data to characterize Waste 1 and Waste 2 as reference materials. Additional intercomparison data will need to be acquired to characterize these materials.

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APPENDIX 1 - RESULTS

Table A1 - Sample 02-1-1 Ampouled Standard - Results in mg/L																
	2101	**2102**	**2103**	**2104**	**2105**	**2106**	**2107**	**2108**	**2109**	**2111**	**2112**	**2113**	**2116**	**2117**	**2118**	**2119**
Aluminum	0.68	0.60	0.54	0.645	0.60	0.57	<1.0	0.31	0.64	0.70	0.60	0.691	0.604	0.620	1.56	0.66
Barium	0.11	0.10	ND	0.0964	0.10	0.10	<1.0	0.049	0.102	0.06	0.10	0.106	0.0996	0.102	0.61	0.098
Beryllium	0.074	0.10	0.093	0.0915	0.10	0.095	0.10	0.062	0.098	0.102	0.10	0.11	0.0987	0.1035	0.101	0.0999
Cadmium	0.11	0.11	0.10	0.103	0.10	0.10	0.10	0.062	0.0985	0.103	0.101	0.112	0.109	0.101	0.10	0.103
Calcium	1.00	1.28	0.40	1.65	0.99	<0.5	1.4	0.76	1.3	1.3	1.30	1.42	1.6	1.32	1.00	1.27
Chromium	0.10	0.11	0.102	0.105	0.10	0.098	0.10	0.06	0.099	0.101	0.11	0.11	0.121	0.096	0.10	0.105
Cobalt	0.10	0.10	0.10	0.102	0.10	0.094	0.10	0.05	0.0961	0.096	0.10	0.11	0.103	0.097	0.10	0.102
Copper	0.085	0.08	0.099	0.074	0.08	0.072	<0.2	0.025	0.0768	0.102	<0.1	0.08	0.076	0.070	0.07	0.079
Iron	1.00	0.96	1.01	1.05	1.02	0.98	0.94	0.58	0.99	0.89	1.00	1.09	1.08	0.971	0.99	0.945
Lead	0.46	0.52	0.50	0.529	0.50	0.50	0.47	0.28	0.509	0.509	0.50	0.547	0.585	0.48	0.51	0.51
Magnesium	2.00	0.47	0.467	0.486	0.50	0.50	0.50	0.25	0.47	0.4	0.50	0.523	0.46	0.49	<1	0.53
Manganese	0.40	0.30	0.30	0.309	0.30	0.29	0.29	0.19	0.292	0.304	0.30	0.328	0.321	0.298	0.25	0.307
Molybdenum	0.39	0.35	0.429	0.411	0.30	0.36	0.32	0.16	0.401	0.393	0.40	0.428	0.391	0.392	0.39	0.29
Nickel	0.39	0.39	0.40	0.413	0.40	0.38	0.38	0.21	0.39	0.411	0.41	0.44	0.435	0.391	0.41	0.40
Strontium	0.39	0.41	0.133	0.403	0.405	0.38	0.38	0.234	0.398	0.392	0.41	0.418	0.389	0.397	0.378	0.386
Titanium	0.38			0.381	0.40	0.400	0.38	0.21				0.423	0.394	0.403	0.40	0.376
Vanadium	0.42	0.38	ND	0.41	0.40	0.39	0.39	0.23	0.395	0.394	0.40	0.426	0.412	0.400	0.39	0.389
Zinc	0.48	0.50	0.51	0.521	0.50	0.52	0.47	0.29	0.5	0.526	0.50	0.561	0.528	0.490	0.52	0.527

Table A1 continued - Sample 02-1-1 Ampouled Standard - Results in mg/L													
	2120	**2121**	**2122**	**2123**	**2124**	**2125**	**2126**	**2128**	**2130**	**2131**	**2132**	**2133-ICP**	**2133-ICP/MS**
Aluminum	0.89	0.65	2.074	0.60	0.03	0.60	0.770	0.133	0.776	0.57	0.75	0.608	0.988
Barium	0.113	0.095	0.392	0.10	0.04	0.093	0.108	0.099	0.095	0.10	0.107	0.093	0.104
Beryllium	0.112	0.099	0.388	0.096	0.01	0.11	0.099	0.099	0.097	0.08	0.0995	0.093	0.091
Cadmium	0.115	0.10	0.409	0.10	0.002	0.098	0.098	0.097	0.097	0.10	0.084	0.094	0.098
Calcium	1.75	1.5	3.291	1.21	0.05	1.10	1.26	2.62	1.02	1.26	1.36	1.35	< 5
Chromium	0.112	0.10	0.395	0.10	0.01	0.10	0.106	0.101	0.101	0.10	0.088	0.09	0.10
Cobalt	0.11	0.10	0.399	0.10	0.02	0.096	0.098	0.096	0.099	0.10	0.089	0.089	0.096
Copper	0.086	0.078	0.291	0.07	0.01	0.075	0.085	0.08	0.08	0.07	0.075	0.113	0.069
Iron	1.16	1.10	3.957	1.00	0.02	1.04	0.978	1.04	1.04	0.91	0.858	0.925	0.580
Lead	0.559	0.52	2.025	0.50	0.03	0.50	0.505	0.491	0.51	0.50	0.421	0.435	0.464
Magnesium	0.58	0.55	1.381	0.50	0.02	0.50	0.42	0.47	0.484	0.48	0.46	0.485	0.496
Manganese	0.336	0.30	0.074	0.295	0.02	0.28	0.294	0.288	0.308	0.30	0.278	0.279	0.264
Molybdenum	0.442	0.37	0.094	0.38	0.02	0.38	0.391	0.385	0.34	0.38	0.32	0.418	0.361
Nickel	0.443	0.40	0.102	0.39	0.05	0.41	0.394	0.389	0.41	0.39	0.351	0.391	0.325
Strontium	0.442	0.38	0.101	0.393	0.02	0.38	0.382	0.394	0.404	0.40	0.347	0.382	0.394
Titanium	0.442	N/A	0.10	0.40		0.39	0.384	0.354	0.402		0.439	0.363	0.223
Vanadium	0.442	0.40	0.097	0.40	0.01	0.38	0.417	0.381	0.401	0.37	0.373	0.369	0.298
Zinc	0.592	0.55	0.131	0.506	0.01	0.55	0.480	0.478	0.508	0.50	0.501	0.472	0.359

Table A2 - Sample 02-1-2
Ampouled Standard - Results in mg/L

	2101	2102	2103	2104	2105	2106	2107	2108	2109	2111	2112	2113	2116	2117	2118	2119
Aluminum	2.1	2.00	1.62	2.12	2.00	1.85	2.00	0.1	2.08	2.13	2.1	2.29	2.12	1.99	2.37	2.16
Barium	0.45	0.39	ND	0.389	0.40	0.38	<1.0	0.006	0.408	0.35	0.4	0.42	0.389	0.403	0.64	0.395
Beryllium	0.31	0.40	0.366	0.37	0.40	0.39	0.38	0.036	0.401	0.406	0.41	0.438	0.398	0.4271	0.40	0.405
Cadmium	0.43	0.42	0.386	0.504	0.41	0.40	0.39	0.02	0.399	0.41	0.407	0.444	0.441	0.401	0.397	0.418
Calcium	3.6	3.79	1.046	4.73	2.96	3.49	3.7	0.44	3.7	3.8	3.9	4.17	3.8	3.95	4.00	3.83
Chromium	0.4	0.41	0.408	0.397	0.40	0.39	0.38	0.02	0.393	0.399	0.41	0.434	0.408	0.394	0.39	0.422
Cobalt	0.39	0.41	0.4	0.389	0.41	0.38	0.37	0.02	0.387	0.38	0.4	0.433	0.405	0.381	0.39	0.412
Copper	0.31	0.30	0.273	0.301	0.31	0.28	0.3	<0.005	0.30	0.303	0.3	0.311	0.306	0.290	0.27	0.309
Iron	4.2	3.79	3.92	4.11	4.00	3.89	3.80	0.4	3.86	4.12	4.1	4.27	4.32	3.948	3.88	4.09
Lead	1.2	2.05	1.96	2.03	2.00	1.89	1.90	0.09	2.01	2.16	1.99	2.18	2.16	1.95	2.02	2.11
Magnesium	5.8	1.48	1.463	1.46	1.49	1.44	1.40	0.034	1.42	1.5	1.5	1.57	1.40	1.48	2.00	1.54
Manganese	0.1	0.08	0.075	0.075	0.08	0.073	0.07	0.008	0.075	0.077	0.08	0.082	0.0838	0.075	0.08	0.079
Molybdenum	0.1	0.12	0.214	0.104	0.1	0.093	0.06	<0.02	0.101	0.099	0.1	0.108	0.099	0.096	0.10	0.085
Nickel	0.1	0.10	0.125	0.103	0.1	0.097	<0.3	0.02	0.099	0.103	0.1	0.11	0.114	0.099	0.10	0.10
Strontium	0.099	0.11	0.031	0.1	0.105	0.10	0.10	0.0071	0.102	0.099	0.1	0.108	0.101	0.102	0.092	0.10
Titanium	0.1			0.095	0.1	0.101	0.10	<0.02				0.107	0.102	0.103	0.10	0.098
Vanadium	0.1	0.10	ND	0.1	0.1	0.092	0.10	0.004	0.101	0.100	0.1	0.108	0.106	0.092	0.10	0.102
Zinc	0.12	0.13	0.157	0.129	0.12	0.13	<0.2	0.01	0.126	0.135	<0.2	0.142	0.142	0.132	0.13	0.136

Table A2 continued - Sample 02-1-2 Ampouled Standard - Results in mg/L													
	2120	**2121**	**2122**	**2123**	**2124**	**2125**	**2126**	**2128**	**2130**	**2131**	**2132**	**2133-ICP**	**2133-ICP/MS**
Aluminum	2.25	2.20	0.60	2.00	15.03	1.95	2.160	0.416	2.04	2.00	2.47	1.941	2.162
Barium	0.408	0.38	0.096	0.38	19.93	0.37	0.414	0.385	0.379	0.40	0.429	0.363	0.392
Beryllium	0.412	0.39	0.095	0.386	5.03	0.41	0.397	0.387	0.387	0.37	0.404	0.361	0.363
Cadmium	0.423	0.41	0.10	0.41	1.00	0.388	0.384	0.38	0.39	0.38	0.348	0.365	0.384
Calcium	3.96	3.90	0.944	3.59	24.96	3.5	3.73	4.28	3.37	3.65	4.11	3.56	< 5
Chromium	0.402	0.40	0.102	0.40	9.95	0.39	0.396	0.388	0.405	0.39	0.371	0.364	0.311
Cobalt	0.402	0.40	0.093	0.38	9.90	0.38	0.394	0.376	0.397	0.39	0.359	0.358	0.386
Copper	0.317	0.29	0.072	0.29	5.02	0.29	0.304	0.296	0.308	0.28	0.332	0.310	0.290
Iron	4.11	4.40	0.927	3.95	9.97	3.92	3.900	4.1	3.97	3.85	4.8	3.706	2.73
Lead	1.93	2.00	0.511	2.00	14.89	1.94	1.960	1.91	2.05	2.00	1.75	1.842	1.878
Magnesium	1.58	1.60	0.351	1.4	10.00	1.4	1.38	1.54	1.4	1.42	1.42	1.378	0.988
Manganese	0.0784	0.08	0.294	0.074	10.10	0.07	0.073	0.075	0.076	0.08	0.0709	0.070	0.056
Molybdenum	0.103	0.095	0.383	0.10	10.00	0.10	0.101	0.097	0.091	0.10	0.08	0.135	0.094
Nickel	0.104	0.10	0.387	0.09	24.92	0.096	0.098	0.106	1.04	0.10	0.091	0.096	0.082
Strontium	0.104	0.10	0.397	0.099	9.97	0.097	0.097	0.102	1.03	0.10	0.089	0.096	0.096
Titanium	0.102	N/A	0.39	0.10		0.10	0.097	0.104	0.097		0.113	0.090	0.056
Vanadium	0.09	0.10	0.394	0.10	4.98	0.094	0.101	0.093	0.094	0.09	0.094	0.090	0.087
Zinc	0.155	0.14	0.511	0.128	5.02	0.14	0.119	0.127	0.126	0.14	0.13	0.125	0.103

Table A3 - Sample 02-1-3 Leachate 1 - Results in mg/L															
	2101	**2102**	**2103**	**2104**	**2105**	**2106**	**2107**	**2108**	**2109**	**2110**	**2111**	**2112**	**2113**	**2116**	**2117**
Aluminum	1.2	1.35	0.625	1.36	1.16	1.14	1.5	1.39	1.32	1.587	1.27	1.4	1.40	1.14	0.18
Barium	0.51	0.48	ND	0.508	0.485	0.48	1.1	0.38	0.47	0.468	0.44	0.5	0.488	0.501	0.474
Beryllium	< 0.0010	<0.005	ND	<0.0002	<0.005	<0.001	<0.01	<0.001	<0.01	0	0.0007	<0.01	<0.010	0.0011	0.0009
Cadmium	0.46	0.43	0.425	0.431	0.46	0.41	0.41	0.39	0.418	0.347	0.358	0.407	0.436	0.419	0.440
Calcium	63	224	207.7	226	229	220	235	231	219	236	251	242	230	238	235
Chromium	3.8	4.33	4.49	4.42	4.26	4.02	4.0	3.53	4.22	4.602	4.40	4.18	4.30	4.36	4.21
Cobalt	< 0.0050	<0.01	0.017	0.0041	<0.01	0.002	<0.02	<0.01	0.002	0.002	0.002	<0.01	<0.015	0.0028	< 0.005
Copper	0.09	0.09	0.114	0.117	0.11	0.092	<0.2	0.082	0.099	0.105	0.098	0.1	0.103	0.111	0.085
Iron	1.4	0.24	0.29	0.27	0.29	0.52	0.34	0.23	0.5	1.14	0.31	0.2	0.275	0.263	0.257
Lead	1.2	4.26	4.33	4.34	4.4	4.10	4.1	3.5	4.15	4.046	4.19	4.18	4.34	4.21	4.07
Magnesium	22	22.9	21.95	23.6	22.4	21	21.1	23.6	21.5	23.0	18.9	22.6	22.2	22.6	22.1
Manganese	1.2	3.59	3.74	3.7	3.64	3.35	3.3	3.09	3.49	3.426	3.45	3.36	3.59	3.76	3.544
Molybdenum	< 0.0050	<0.02	ND	0.003	<0.02	<0.001	<0.05	<0.02	<0.01	0	0.001	0.02	<0.015	<0.001	0.008
Nickel	0.015	<0.02	0.025	0.0095	0.04	0.010	<0.3	<0.02	<0.05	0.016	0.014	<0.02	<0.015	0.011	0.009
Strontium	0.43	0.48	0.285	0.509	0.47	0.45	0.42	0.428	0.45	0.448	0.412	0.48	0.455	0.483	0.462
Titanium	0.02			<0.001	<0.01	<0.002	<0.05	<0.02		0.019			<0.005	0.0017	< 0.003
Vanadium	< 0.010	0.04	ND	<0.0008	<0.005	<0.001	<0.02	<0.002	<0.005	0.002	<0.001	<0.01	<0.015	<0.001	< 0.005
Zinc	0.16	0.17	0.185	0.173	0.19	0.14	0.75	0.14	0.17	0.15	0.152	<0.2	0.175	0.158	0.162

Table A3 continued - Sample 02-1-3 Leachate 1 - Results in mg/L															
	2118	2119	2120	2121	2122	2123	2124	2125	2126	2128	2130	2131	2132	2133-ICP	2133-ICP/MS
Aluminum	1.07	1.42	1.52	1.50	NA	1.4	1.44	1.0	1.23	0.156	1.31	1.2	1.29	1.315	1.384
Barium	0.39	0.451	0.523	0.45	0.487	0.48	0.49	0.5	0.471	0.41	0.472	0.48	0.442	0.483	0.477
Beryllium	<0.002	0.0008	0.001	0.001	<0.007	<0.005	<0.01	0.003	0.001<W	<0.01	0.001	< 0.01	0.0009	0.001	< 0.001
Cadmium	0.389	0.409	0.462	0.42	0.42	0.44	0.459	0.4	0.353	0.401	0.425	0.40	0.381	0.420	0.411
Calcium	243	233	230	230	220	217	219	210	204	210.2	217	226	244	223	143
Chromium	3.84	4.15	4.38	4.0	4.15	4.22	4.42	4.0	3.79	4.45	4.39	4.1	4.52	4.168	3.660
Cobalt	<0.01	<0.005	0.0019	<0.005	<0.01	<0.01	<0.02	<0,005	0.001<W	<0.01	0.003	< 0.01	0.002	< 0.005	0.002
Copper	0.09	0.108	0.104	0.091	0.11	0.11	0.07	0.10	0.101	0.115	0.134	0.09	0.093	0.106	0.092
Iron	0.24	0.268	0.28	0.22	0.24	0.25	0.28	0.6	0.299	0.267	0.275	0.24	0.235	0.276	< 0.5
Lead	3.72	4.17	4.11	4.1	4.22	4.18	4.44	4.0	3.68	4.48	4.4	4.1	3.8	4.167	4.116
Magnesium	21	21.5	24.1	23	23.5	21.1	23.2	23	19.9	25	22.6	23	20.5	22.1	8.70
Manganese	3.11	3.43	3.64	3.4	NA	3.45	3.65	3.0	3.13	3.6	3.61	3.2	3.19	3.475	3.154
Molybdenum	<0.01	<0.005	0.0002	<0.006	NA	<0.03	<0.02	0.01	0.001<W	<0.01	0.013	< 0.02	<0.01	0.067	< 0.001
Nickel	<0.01	0.01	0.013	<0.01	0.03	<0.05	<0.05	0.009	0.016<T	0.013	0.017	< 0.02	0.009	0.023	0.011
Strontium	0.386	0.419	0.526	0.42	0.464	0.455	0.47	0.4	0.405	0.394	0.467	0.40	0.394	0.460	0.453
Titanium	<0.01	<0.003	0.0017	N/A	NA	<0.01		<0,05	0.001<W	<0.01	<0.02		<0.001	< 0.005	0.014
Vanadium	<0.01	<0.005	<0.001	<0.003	<0.006	<0.03	<0.01	<0,007	0.001<W	<0.01	<0.02	< 0.03	0.013	< 0.005	< 0.005
Zinc	0.14	0.166	0.20	0.17	0.175	0.171	0.17	0.2	0.17	0.306	0.166	0.17	0.162	0.276	0.102

Table A4 - Sample 02-1-4 Leachate 2 - Results in mg/L																
	2101	**2102**	**2103**	**2104**	**2105**	**2106**	**2107**	**2108**	**2109**	**2110**	**2111**	**2112**	**2113**	**2116**	**2117**	**2118**
Aluminum	0.13	0.12	0.051	0.076	0.08	0.12	<1.0	0.17	0.15	0.253	0.17	<0.1	<0.250	0.093	< 0.02	0.10
Barium	0.24	0.23	ND	0.082	0.24	0.25	<1.0	0.18	0.26	0.257	0.19	0.20	0.254	0.243	0.242	0.16
Beryllium	< 0.0010	<0.005	ND	0.246	<0.005	<0.001	<0.01	<0.001	<0.01	0	<0.0005	<0.01	<0.010	<0.0005	0.0002	<0.002
Cadmium	0.63	0.58	0.593	0.598	0.62	0.46	0.57	0.61	0.619	0.469	0.469	0.513	0.633	0.539	0.634	0.50
Calcium	54.0	183.0	188.5	198.0	188.0	180.0	191.0	196.0	184.0	185.0	179.0	193.0	198.0	193	197	179.0
Chromium	5.3	5.67	6.53	5.91	5.84	5.80	5.3	4.34	6.13	6.384	5.82	5.52	6.21	6.20	6.01	4.65
Cobalt	< 0.0050	<0.01	0.017	0.0023	<0.01	<0.001	<0.02	<0.01	<0.001	0.001	0.001	<0.01	<0.015	0.0024	< 0.005	<0.01
Copper	0.095	0.09	0.095	0.097	0.10	0.080	<0.2	0.062	0.092	0.10	0.104	<0.1	0.095	0.098	0.070	0.07
Iron	1.6	<0.1	0.114	0.095	0.11	0.38	0.29	0.06	0.50	0.863	1.12	<0.1	<0.100	0.077	0.084	0.07
Lead	1.5	5.71	6.25	6.07	6.1	5.82	5.6	3.85	6.01	5.778	5.61	5.38	6.41	5.42	5.42	4.40
Magnesium	15.0	13.9	14.48	14.2	13.7	12.7	12.9	8.52	5.6	14.3	11.2	13.8	14.5	13.9	13.8	11.0
Manganese	0.85	0.76	0.96	0.793	0.80	0.79	0.73	0.60	0.82	0.808	0.747	0.71	0.852	0.798	0.831	0.63
Molybdenum	< 0.0050	<0.02	ND	<0.001	<0.02	<0.001	<0.05	<0.02	<0.01	1.0	<0.001	<0.01	<0.015	<0.001	0.010	<0.01
Nickel	0.01	<0.02	0.025	0.0047	0.05	0.005	<0.3	<0.02	<0.05	0.011	0.005	<0.02	<0.015	0.005	< 0.008	<0.01
Strontium	0.36	0.40	0.228	0.423	0.395	0.40	0.35	0.306	0.38	0.384	0.303	0.39	0.402	0.398	0.400	0.266
Titanium	0.02			<0.001	<0.01	0.005	<0.05	<0.02		0.021			<0.005	0.0055	< 0.003	<0.01
Vanadium	< 0.010	0.04	ND	<0.0008	<0.005	<0.001	<0.02	<0.002	<0.005	0.002	<0.001	<0.01	<0.015	<0.001	< 0.005	<0.01
Zinc	0.38	0.44	0.51	0.464	0.49	0.31	1.0	0.43	0.46	0.352	0.397	0.40	0.485	0.413	0.444	0.35

Table A4 continued - Sample 02-1-4
Leachate 2 - Results in mg/L

	2119	2120	2121	2122	2123	2124	2125	2126	2128	2130	2131	2132	2133-ICP	2133-ICP/MS
Aluminum	0.32	0.21	0.34	NA	<0.4	0.26	0.10	0.35	0.148	0.171	0.20	0.12	0.101	0.115
Barium	0.221	0.252	0.22	0.239	0.23	0.24	0.20	0.235	0.209	0.216	0.23	0.18	0.228	0.234
Beryllium	<0.0002	<0.001	<0.001	<0.007	<0.01	<0.01	<0,001	0.001<W	<0.01	<0.001	< 0.01	<0.0006	< 0.001	< 0.001
Cadmium	0.554	0.624	0.56	0.58	0.61	0.652	0.50	0.504	0.616	0.576	0.56	0.48	0.538	0.563
Calcium	188.0	198.0	190.0	183.0	178.0	182.0	190.0	172	274.9	178.0	185.0	0.19	173	123.0
Chromium	5.57	6.33	5.40	5.74	5.74	6.09	6.0	5.31	6.47	5.58	5.5	5.9	5.462	4.996
Cobalt	<0.005	0.0004	<0.005	<0.01	<0.02	<0.02	<0,005	0.001<W	<0.01	0.005	< 0.01	0.001	0.011	< 0.001
Copper	0.098	0.088	0.085	0.094	0.08	0.06	0.08	0.092	0.112	0.124	0.07	0.075	0.099	0.092
Iron	0.084	0.14	0.067	0.05	0.07	0.12	0.30	0.253	0.115	0.088	0.08	0.068	0.068	< 0.5
Lead	5.66	6.40	5.60	5.69	5.70	6.33	6.0	5.3	6.48	5.77	5.60	4.63	5.472	5.956
Magnesium	13.0	14.3	14.0	14.0	13.1	14.9	15.0	12.6	16.7	15.3	14.0	11.1	12.9	5.38
Manganese	0.748	0.823	0.73	NA	0.76	0.82	0.70	0.716	0.893	0.795	0.74	0.648	0.743	0.606
Molybdenum	<0.005	<0.0002	<0.006	NA	<0.06	<0.02	<0,009	0.001<W	<0.01	0.01	< 0.02	<0.01	0.042	< 0.001
Nickel	0.01	0.008	<0.01	<0.02	<0.1	<0.05	0.003	0.008<T	0.019	0.007	< 0.02	0.003	0.005	0.027
Strontium	0.348	0.426	0.35	0.386	0.37	0.39	0.40	0.338	0.37	0.361	0.32	0.29	0.371	0.383
Titanium	<0.003	0.0044	N/A	NA	<0.02		<0,05	0.003<T	<0.01	<0.02		<0.001	< 0.005	0.015
Vanadium	<0.005	<0.001	<0.003	<0.006	<0.06	<0.01	<0,007	0.001<W	<0.01	<0.02	< 0.03	0.012	< 0.005	< 0.005
Zinc	0.438	0.47	0.44	0.437	0.87	0.47	0.40	0.473	0.415	0.436	0.42	0.416	0.068	0.237

Table A5 - Sample 02-1-5 Waste 1 - Results in mg/L																
	2101	**2102**	**2103**	**2104**	**2105**	**2106**	**2107**	**2108**	**2109**	**2110**	**2111**	**2112**	**2113**	**2116**	**2117**	**2118**
Aluminum	1.2	0.09	ND	<0.025	0.29	<0.01	<1.0	0.08	0.08	0.103	0.10	<0.1	<0.250	0.031	< 0.02	<0.05
Barium	0.16	0.29	ND	0.346	0.305	0.64	<1.0	0.20	0.87	0.337	0.15	0.40	0.353	0.381	0.414	0.24
Beryllium	< 0.0010	<0.005	0.003	<0.001	<0.005	<0.001	<0.01	<0.001	<0.01	0	<0.0005	<0.01	<0.010	<0.0005	< 0.0002	<0.002
Cadmium	0.008	0.03	0.298	0.0781	0.10	0.39	1.4	<0.005	6.60	0.018	<0.001	0.336	0.517	0.680	2.39	0.065
Calcium	730	1730	2923.1	2000	1810	1720	2040	1470	2370	1946	641	1510	1780	1930	2020	2160
Chromium	0.80	0.35	0.136	0.244	0.24	0.13	0.12	0.66	0.16	0.461	0.841	0.15	0.158	0.181	0.100	0.21
Cobalt	< 0.0050	<0.01	0.081	<0.0004	0.03	0.020	<0.02	<0.01	0.02	0.003	<0.001	<0.01	<0.015	0.0019	0.005	<0.01
Copper	0.035	<0.01	0.071	<0.0008	<0.01	0.034	<0.2	0.006	1.01	0.03	0.012	<0.1	<0.010	0.013	0.048	<0.01
Iron	0.40	<0.1	0.076	0.037	<0.02	2.35	0.35	<0.02	0.90	5.188	0.06	<0.1	<0.100	0.014	0.021	0.02
Lead	0.055	<0.02	0.357	0.055	0.20	0.24	0.88	<0.02	11.9	0.027	0.008	0.13	0.075	0.411	1.35	<0.05
Magnesium	0.60	80.5	89.94	94.1	90	71	110	27.8	256	81.0	1.0	46.8	96.2	110	102	74
Manganese	< 0.050	<0.01	1.33	0.0418	0.05	0.34	1.5	<0.002	14.7	0.007	0.002	0.24	0.334	1.10	2.248	0.04
Molybdenum	2.6	0.34	0.143	0.234	0.21	0.074	<0.05	1.68	0.03	0.685	2.48	0.09	0.086	0.10	0.035	0.19
Nickel	0.02	<0.02	0.125	<0.0005	<0.02	0.037	<0.3	<0.03	0.63	0.057	<0.002	0.03	0.31	0.034	0.116	<0.01
Strontium	0.53	0.80	0.58	0.975	1.04	1.02	1.1	0.636	1.41	1.103	0.357	0.84	1.10	1.11	0.908	0.76
Titanium	< 0.01			<0.001	<0.01	<0.002	<0.05	<0.02		0.009			<0.005	<0.004	< 0.003	<0.01
Vanadium	< 0.010	0.16	ND	<0.0006	<0.005	0.002	0.02	<0.002	<0.005	0.004	<0.001	<0.01	<0.015	<0.001	< 0.005	<0.01
Zinc	0.30	0.10	8.21	1.61	2.33	16	66.9	0.05	2120	0.313	0.057	12.2	16.9	23.7	175	1.15

Table A5 continued - Sample 02-1-5 Waste 1 - Results in mg/L														
	2119	**2120**	**2121**	**2122**	**2123**	**2124**	**2125**	**2126**	**2128**	**2130**	**2131**	**2132**	**2133-ICP**	**2133-ICP/MS**
Aluminum	0.054	0.08	<0.38	NA	<5	0.29	<0,08	0.005<w	<0.01	<0.05	< 0.02	2.60	0.013	< 0.02
Barium	0.289	0.61	0.44	0.202	<3	0.37	0.30	0.352	0.305	0.264	0.45	1.05	0.324	0.345
Beryllium	0.0002	<0.001	<0.001	<0.007	<0.03	<0.01	<0,01	0.005<T	<0.01	<0.001	< 0.01	<0.005	< 0.001	< 0.001
Cadmium	0.11	4.14	2.30	<0.01	3.51	0.244	0.30	0.233	0.086	0.058	0.44	8.63	0.291	0.315
Calcium	1718	2100	2000	825	1830	1830	1800	1730	2376	1100	1800	3330	1853	1460
Chromium	0.221	0.109	0.072	0.81	<0.3	0.19	0.20	0.225	0.295	0.229	0.16	0.13	0.160	0.166
Cobalt	<0.005	0.0109	0.006	<0.01	<0.05	<0.02	<0,05	0.028	<0.01	<0.01	< 0.01	0.02	< 0.005	< 0.001
Copper	<0.005	0.126	0.037	0.018	0.07	<0.01	<0,03	0.058	0.019	<0.01	0.02	5.62	0.001	0.012
Iron	<0.005	0.82	0.006	<0.01	<0.2	<0.02	4.10	0.207	<0.01	<0.01	0.02	0.21	0.160	< 0.5
Lead	0.06	3.69	1.60	0.03	2.50	<0.03	0.15	0.29	0.18	0.038	0.20	27.6	0.236	0.244
Magnesium	79.61	153	91	9.04	106	99.6	91	90.9	108.3	77.5	86	362	97.3	54.6
Manganese	0.05	4.18	1.60	NA	3.0	0.15	0.30	0.186	0.034	0.033	0.25	28.2	0.249	0.238
Molybdenum	0.1754	0.0331	0.012	NA	<1	0.13	0.10	0.169	0.25	0.238	0.09	<0.1	0.226	0.123
Nickel	<0.008	0.286	0.09	<0.02	<0.3	<0.05	<0,02	0.002<W	<0.01	<0.01	0.03	0.98	0.018	< 0.003
Strontium	0.961	1.48	1.10	0.574	1.20	1.03	1.0	1.0	1.05	0.902	0.90	1.69	1.148	1.141
Titanium	<0.003	0.0038	N/A	NA	<0.3		<0,5	0.001<W	<0.01	<0.02		<0.01	< 0.005	< 0.001
Vanadium	<0.005	0.007	0.007	<0.006	<0.2	<0.01	<0,07	0.003<T	<0.01	<0.02	< 0.03	0.07	< 0.005	< 0.005
Zinc	2.94	487	170	0.248	452	4.21	8.10	6.88	2.52	0.423	20	4100	9.182	10.36

Table A6 - Sample 02-1-6 Waste 2 - Results in mg/L																
	2101	**2102**	**2103**	**2104**	**2105**	**2106**	**2107**	**2108**	**2109**	**2110**	**2111**	**2112**	**2113**	**2116**	**2117**	**2118**
Aluminum	0.92	0.05	ND	<0.025	0.28	<0.01	<1.0	0.08	0.06	0.068	0.07	<0.1	<0.250	0.029	< 0.02	<0.05
Barium	0.19	0.35	ND	0.438	0.375	0.07	<1.0	0.22	1.14	0.377	0.17	0.4	0.401	0.414	0.618	0.30
Beryllium	< 0.0010	<0.005	0.003	<0.0002	<0.005	<0.001	<0.01	<0.001	<0.01	0	<0.0005	<0.01	<0.010	<0.0005	< 0.0002	<0.002
Cadmium	0.007	0.3	0.801	0.756	0.67	1.14	<0.1	<0.005	6.95	0.104	<0.001	0.218	1.12	1.13	3.93	0.538
Calcium	780	1800	1769.2	1940	1770	1620	1750	1510	2380	1869	676	1320	1670	1890	1780	2250
Chromium	0.8	0.19	0.193	0.103	0.12	0.090	0.20	0.53	0.08	0.303	0.746	0.16	0.104	0.157	0.070	0.11
Cobalt	< 0.0050	<0.01	0.083	<0.0004	0.03	0.006	<0.02	<0.01	0.021	0.003	<0.001	<0.01	<0.015	0.0025	0.011	<0.01
Copper	0.04	<0.01	0.063	<0.0008	<0.01	0.049	<0.2	0.011	1.19	0.025	0.013	<0.1	0.012	0.018	0.156	<0.01
Iron	0.4	<0.1	0.083	0.036	<0.02	2.17	<0.1	<0.02	0.80	5.916	0.04	<0.1	<0.100	0.012	0.024	0.02
Lead	0.11	0.05	0.714	0.483	0.5	0.83	<0.1	<0.02	15.6	0.109	0.011	0.07	0.243	0.746	3.91	0.27
Magnesium	2.4	102	104.9	132	108	93	87.3	44.8	277	105	1.3	53	110	117	129	86
Manganese	0.05	0.38	0.21	1.55	1.1	1.90	<0.05	<0.002	19.6	0.126	<0.001	0.42	1.72	1.6	11.32	0.81
Molybdenum	2.4	0.07	0.286	0.088	0.1	0.040	0.32	1.4	0.03	0.268	2.48	0.12	0.049	0.088	0.020	0.06
Nickel	0.025	<0.02	0.10	0.0317	0.05	0.073	<0.3	<0.03	0.64	0.061	<0.002	0.02	0.061	0.054	0.302	0.02
Strontium	0.54	0.83	0.70	0.95	1.02	1.06	0.92	0.643	1.3	1.093	0.366	0.75	1.04	1.1	0.792	0.767
Titanium	< 0.01			<0.001	<0.01	<0.002	<0.05	<0.02		0.011			<0.005	<0.004	< 0.003	<0.01
Vanadium	< 0.010	0.15	ND	<0.0006	<0.005	0.002	<0.02	<0.002	<0.005	0.004	<0.001	<0.01	<0.015	<0.001	< 0.005	<0.01
Zinc	0.42	5.67	27.14	29	20.8	42	1.1	0.04	2140	1.803	0.046	4.8	42.9	46.4	437	19.4

Table A6 continued - Sample 02-1-6 Waste 2 - Results in mg/L														
	2119	**2120**	**2121**	**2122**	**2123**	**2124**	**2125**	**2126**	**2128**	**2130**	**2131**	**2132**	**2133-ICP**	**2133-ICP/MS**
Aluminum	0.044	0.07	<0.38	NA	<5	0.25	<0,08	0.005<W	<0.01	0.05	< 0.02	1.30	0.008	< 0.02
Barium	0.375	0.777	0.55	0.192	<3	0.44	0.4	0.412	0.327	0.352	0.57	1.24	0.417	0.443
Beryllium	<0.0002	<0.001	<0.001	<0.007	<0.03	<0.01	<0,01	0.002<T	<0.01	<0.001	< 0.01	<0.005	< 0.001	< 0.001
Cadmium	1.01	4.64	3.1	<0.01	3.31	1.02	1.0	1.01	0.40	0.693	1.4	8.04	1.177	1.133
Calcium	1651	2010	1900	356	1760	1740	1700	1680	2452	997	1720	3020	1800	1510
Chromium	0.106	0.072	0.056	0.97	<0.3	0.11	0.1	0.136	0.169	0.115	0.11	0.15	0.175	0.126
Cobalt	<0.005	0.0101	0.006	<0.01	<0.05	<0.02	<0,05	0.005<T	<0.01	<0.01	< 0.01	<0.01	< 0.005	0.002
Copper	0.01	0.168	0.041	0.008	0.05	0.01	<0,03	0.046	0.025	<0.01	0.03	1.79	0.009	0.023
Iron	<0.005	<0.05	<0.005	<0.01	<0.2	<0.02	4.1	0.359	<0.01	<0.01	0.02	10.0	0.175	< 0.5
Lead	0.63	6.41	2.8	0.14	2.4	0.14	0.6	0.735	0.443	0.401	0.75	15.0	0.933	0.972
Magnesium	102.8	147	110	0.15	122	120	120	105	142.3	87	114	310	121	67.9
Manganese	1.615	7.21	3.9	NA	4.3	1.98	1.8	1.61	0.652	1.032	2.0	21.8	2.425	2.261
Molybdenum	0.0446	0.0235	0.008	NA	<1	0.05	<0,1	0.078	0.107	0.061	0.07	0.1	0.153	0.051
Nickel	0.053	0.273	0.13	<0.02	<0.3	0.06	0.1	0.016<T	0.024	0.036	0.08	0.73	0.067	0.045
Strontium	0.944	1.18	1.1	0.32	1.1	1.04	1.0	0.983	1.01	0.883	0.90	1.47	1.120	1.112
Titanium	<0.003	0.0034	N/A	NA	<0.3		<0,5	0.001<W	<0.01	<0.02		<0.01	< 0.005	< 0.001
Vanadium	<0.005	0.009	0.008	<0.006	<0.2	<0.01	<0,07	0.016	<0.01	<0.02	< 0.03	0.06	< 0.005	< 0.005
Zinc	45.5	675	270	0.174	318	33.2	44	42.7	19.0	2.44	80	3340	53.72	61.39

APPENDIX 2 - GRAPHS

Figure A2-1: Example of K-S Graph for Standards

TCLP ILS STUDY: K-S PROCEDURE
STANDARDS: CADMIUM

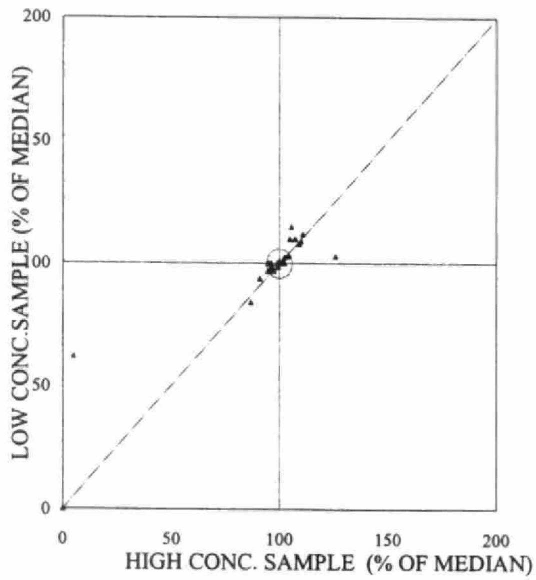


Figure A2-2: Example of K-S Graph for Leachates

TCLP ILS STUDY: K-S PROCEDURE
LEACH: CADMIUM

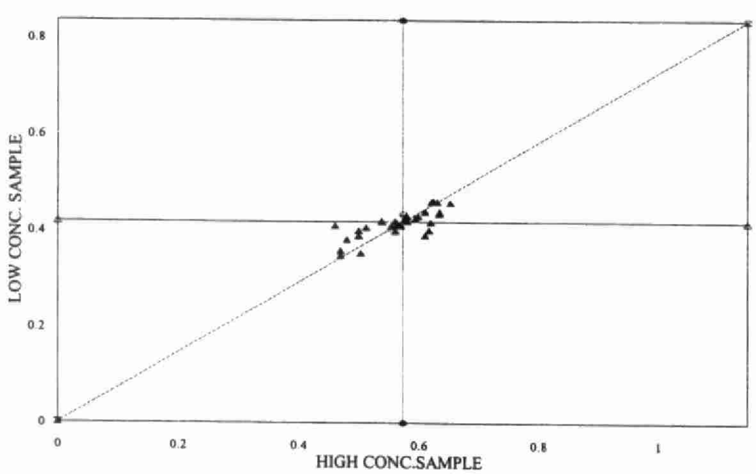


Figure A2-3: Example of Box & Whiskers Plot for Solid Waste

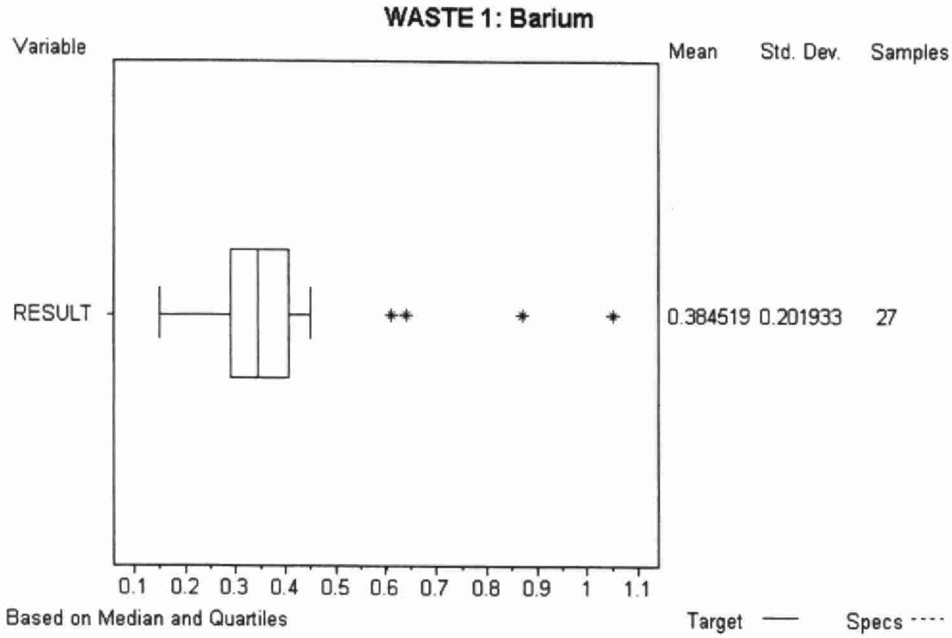
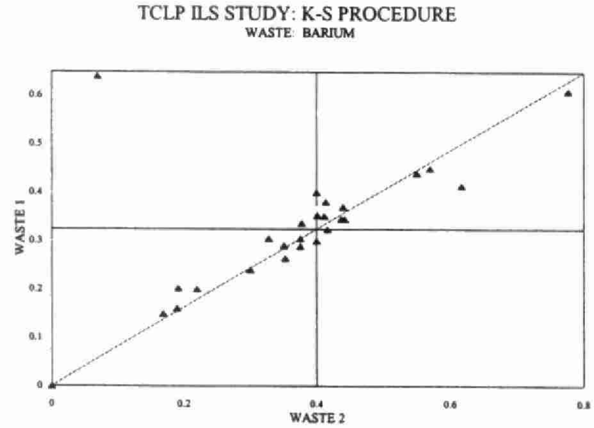
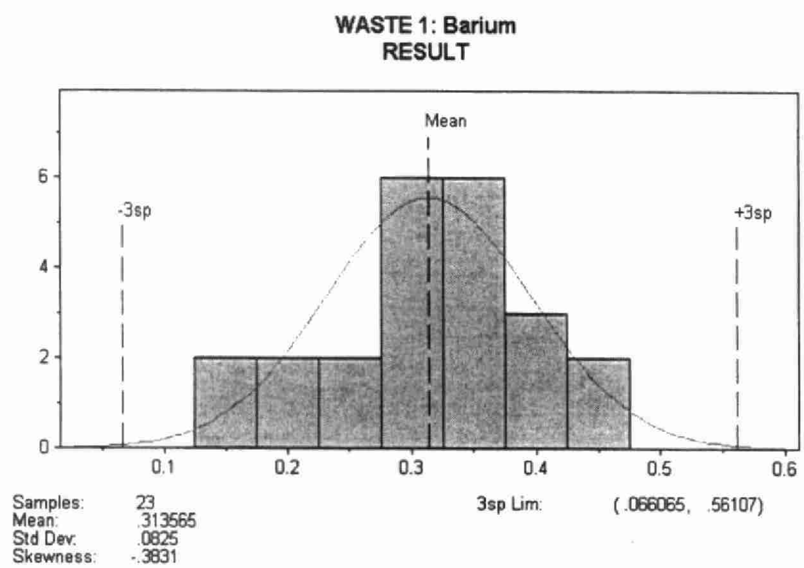


Figure A2-4: Barium in Solid Wastes





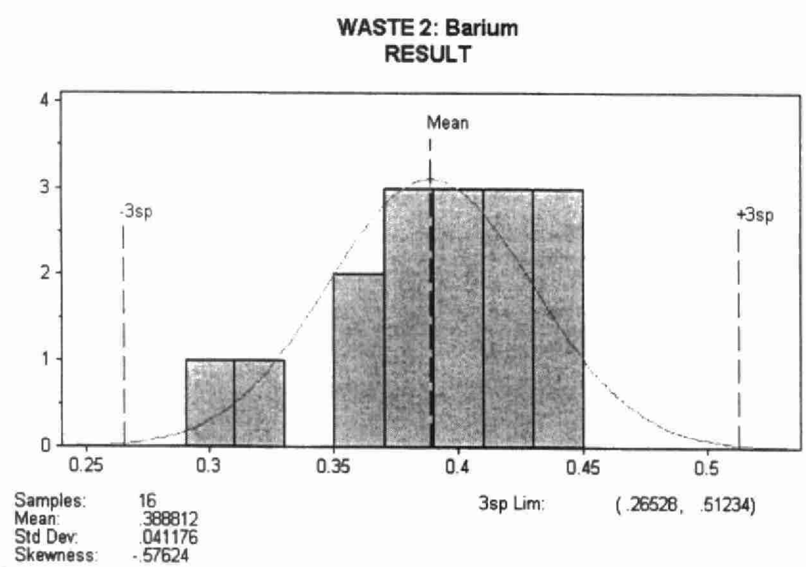
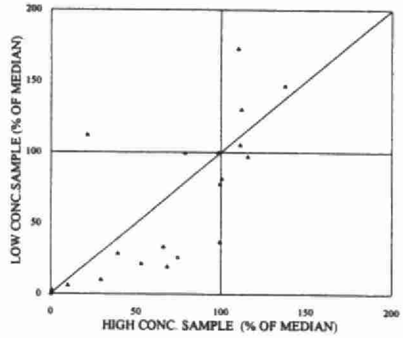
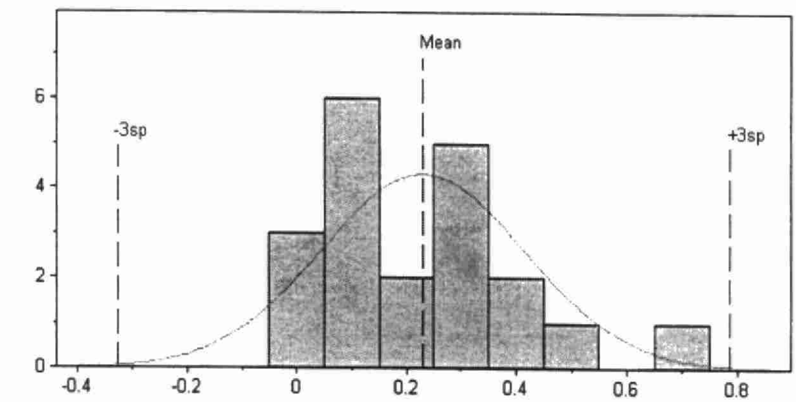


Figure A2-5: Cadmium in Solid Wastes

K-S PROCEDURE: WASTE 1 VS WASTE 2
CADMIUM



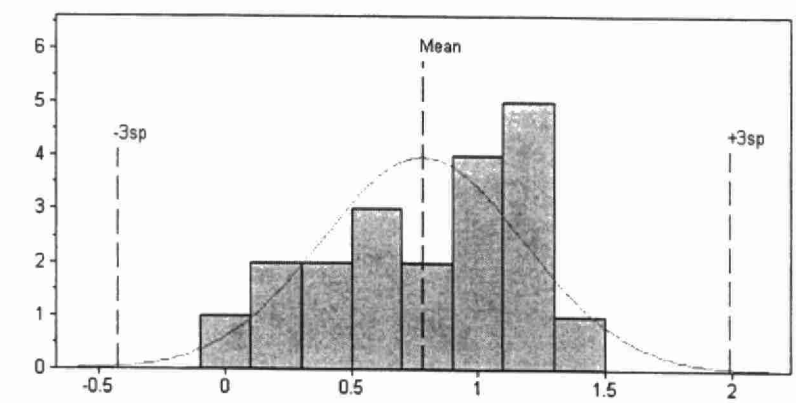
WASTE 1: Cadmium
RESULT



Samples: 20
Mean: .229855
Std Dev: .18533
Skewness: .70092

3sp Lim: (-.32615, .78586)

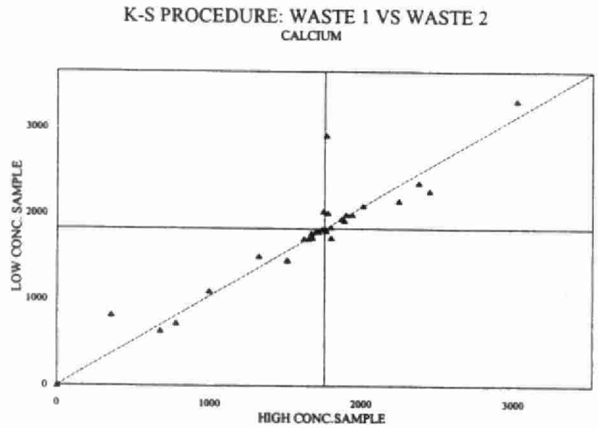
WASTE 2: Cadmium
RESULT

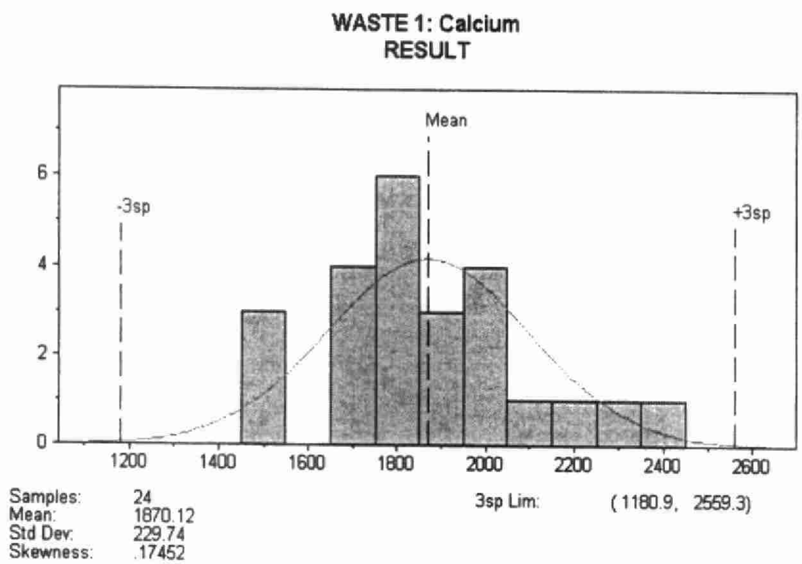


Samples: 20
Mean: .78135
Std Dev: .402
Skewness: -.51522

3sp Lim: (-.42465, 1.9874)

Figure A2-6: Calcium in Solid Wastes





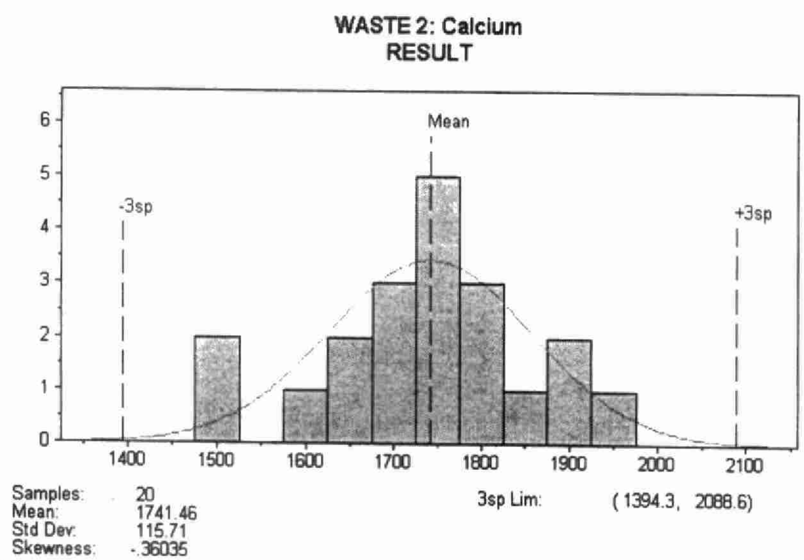
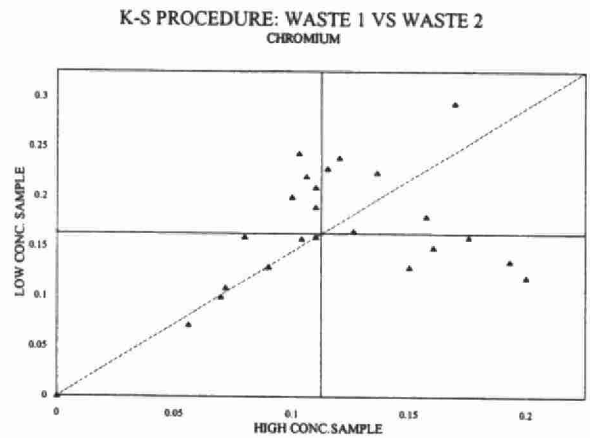
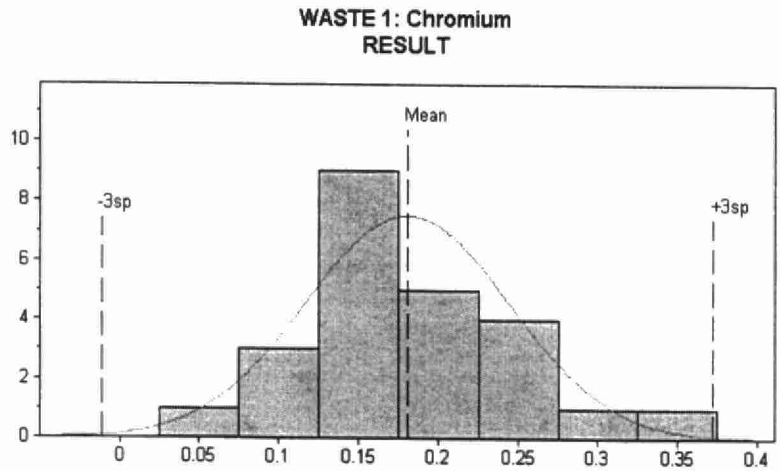


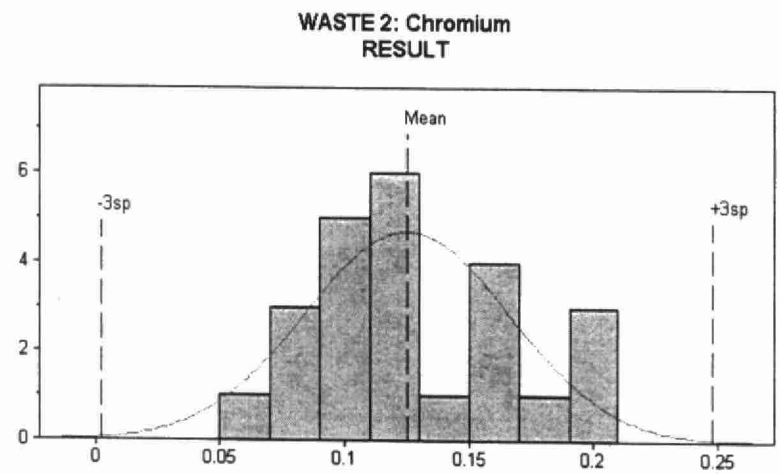
Figure A2-7: Chromium in Solid Wastes





Samples: 24
Mean: .180625
Std Dev: .063841
Skewness: .7348

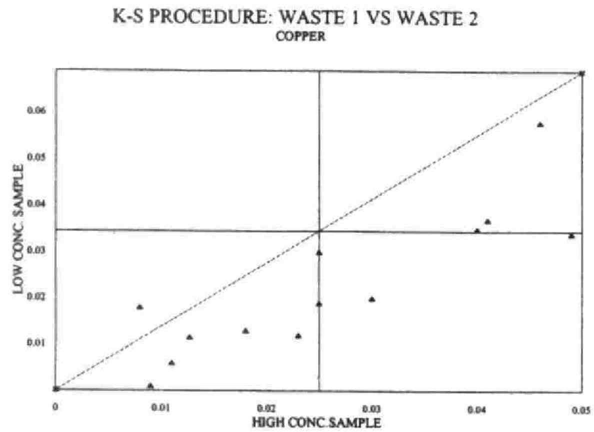
3sp Lim: (-.010897, .37215)

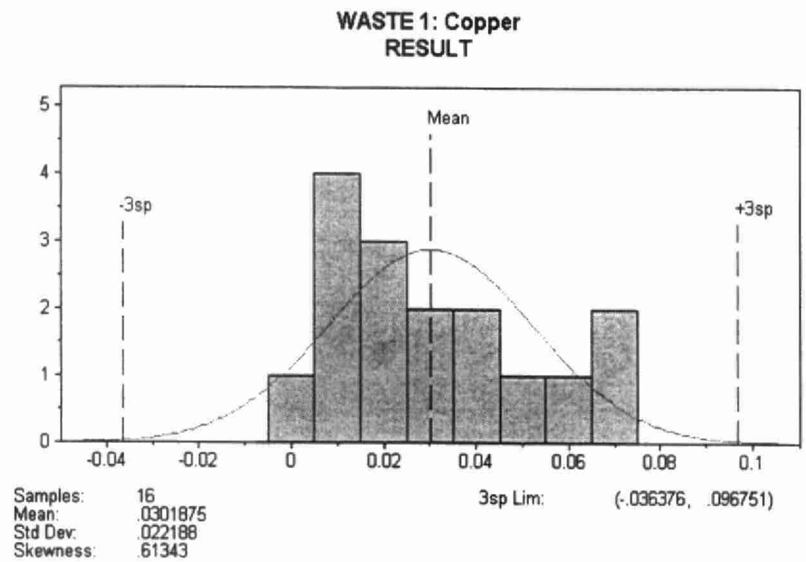


Samples: 24
Mean: .125083
Std Dev: .040893
Skewness: .307

3sp Lim: (.0024037, .24776)

Figure A2-8: Copper in Solid Wastes





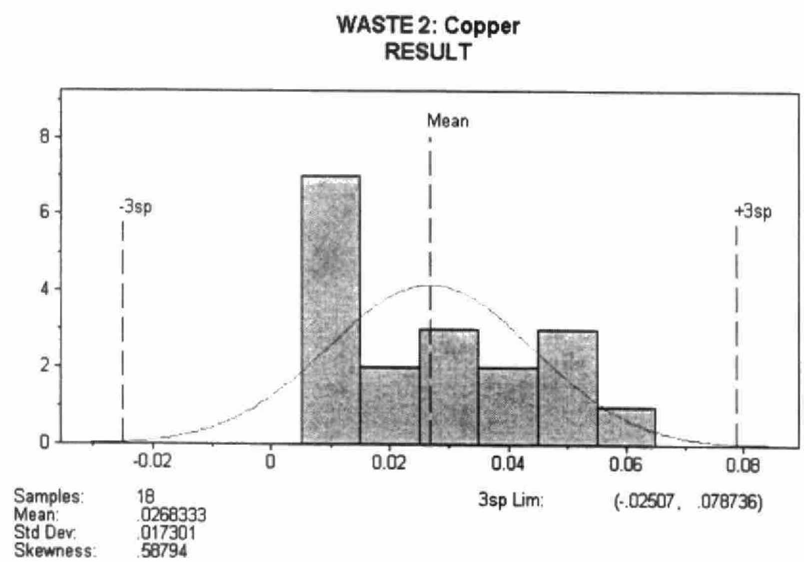
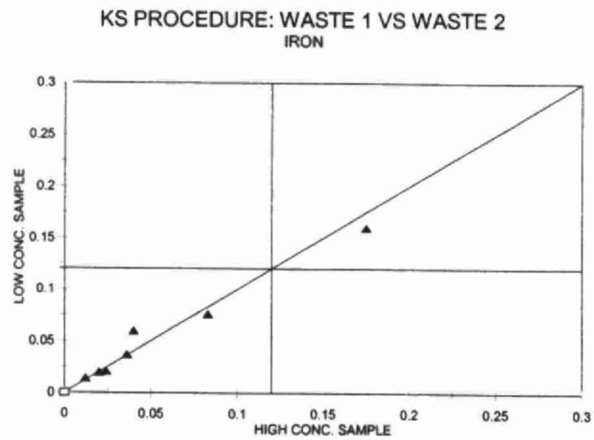


Figure A2-9: Iron in Solid Wastes



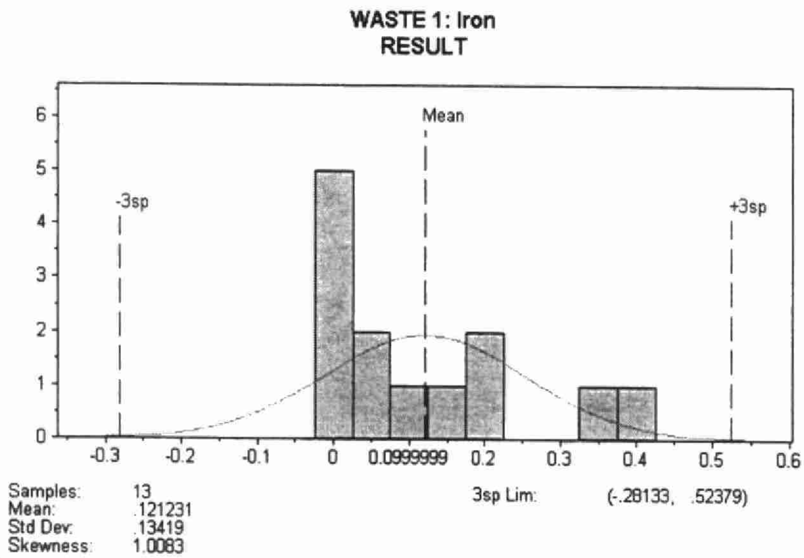
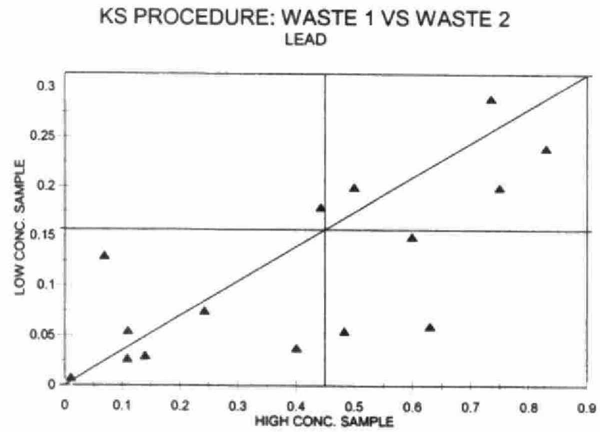
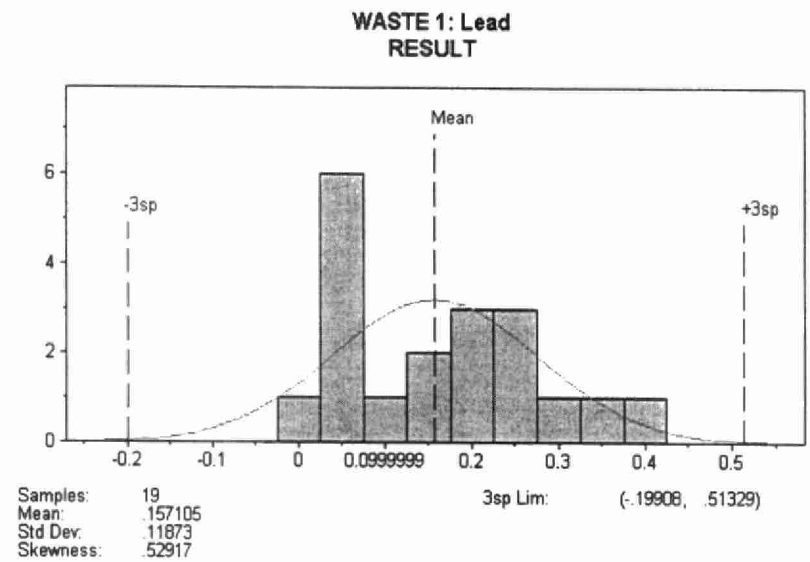


Figure A2-10: Lead in Solid Wastes





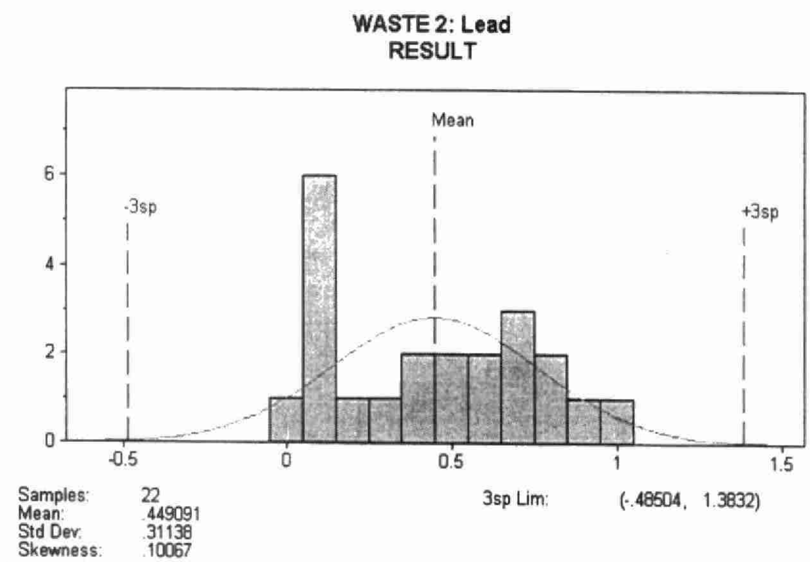
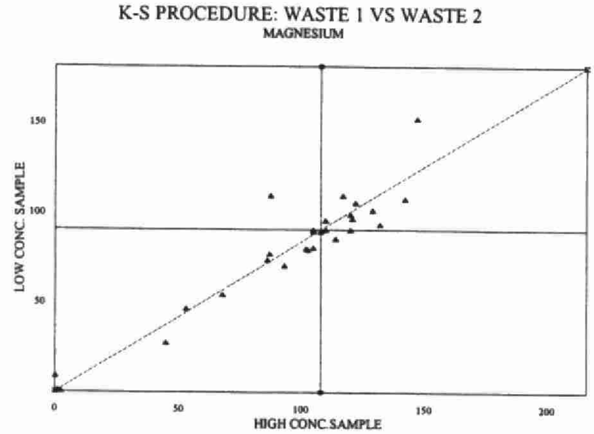
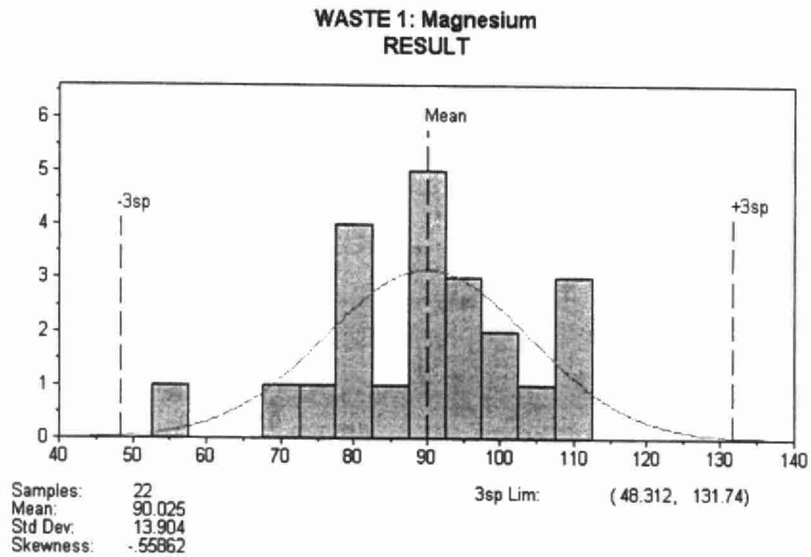


Figure A2-11: Magnesium in Solid Wastes





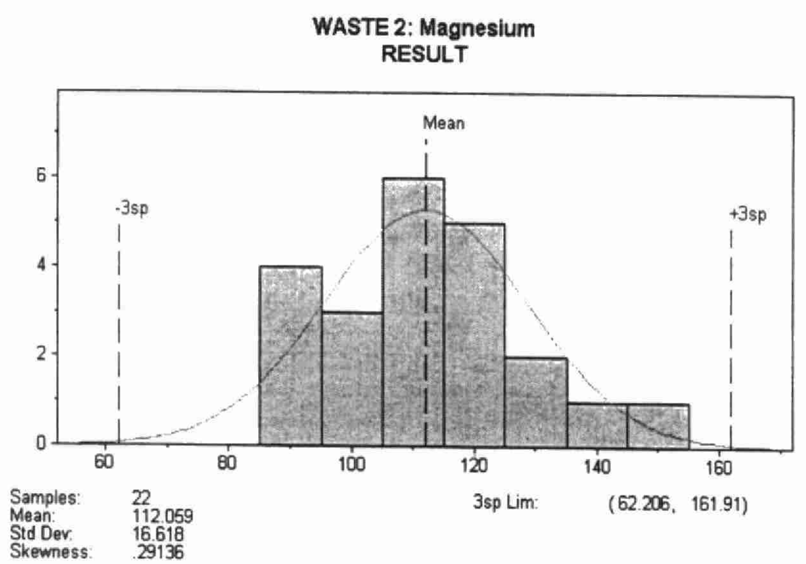
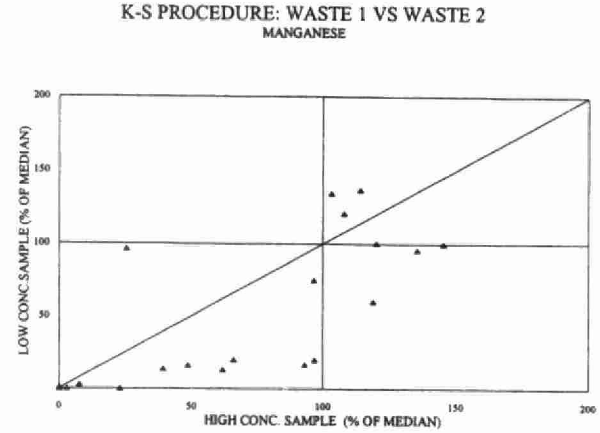
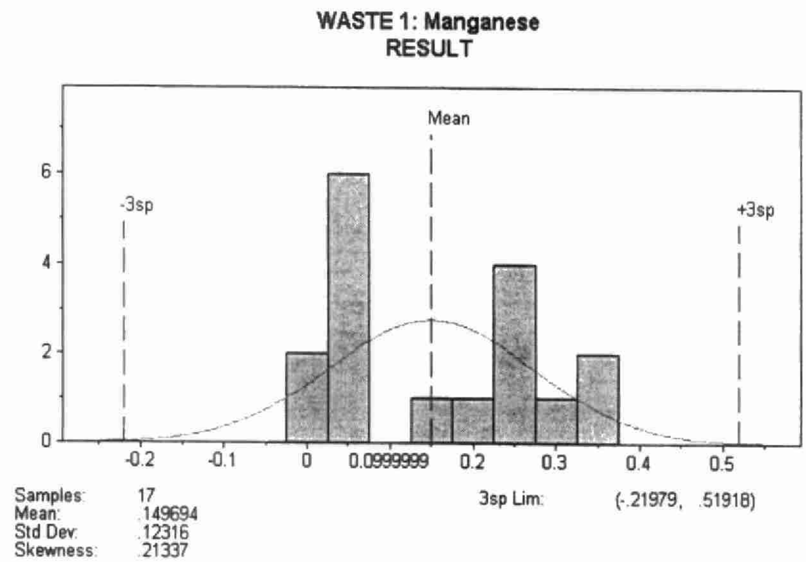


Figure A2-12: Manganese in Solid Wastes





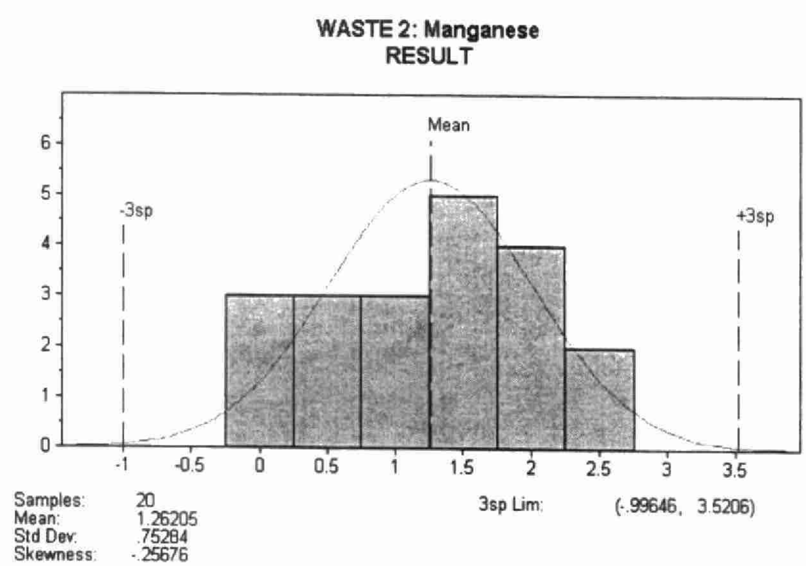
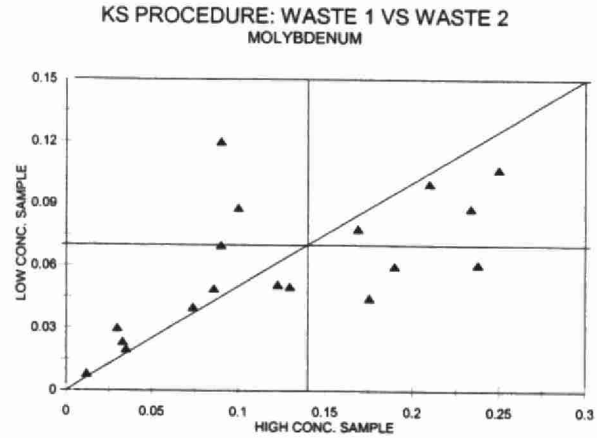
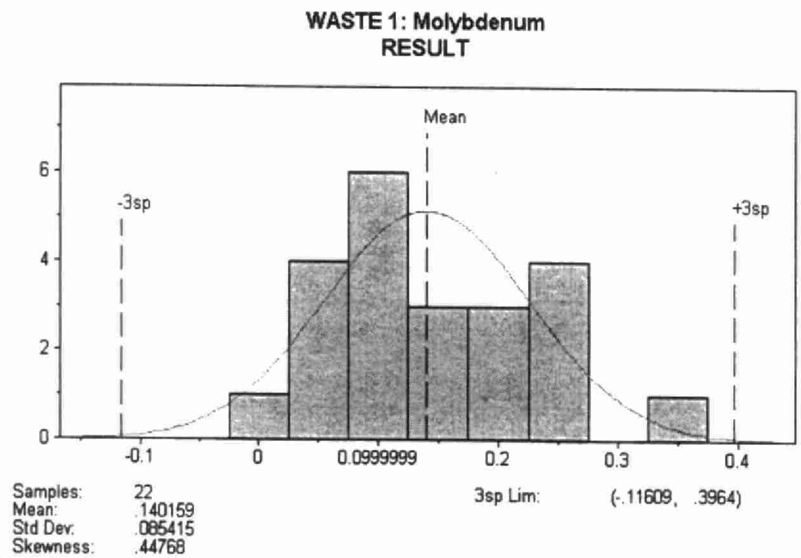


Figure A2-13: Molybdenum in Solid Wastes





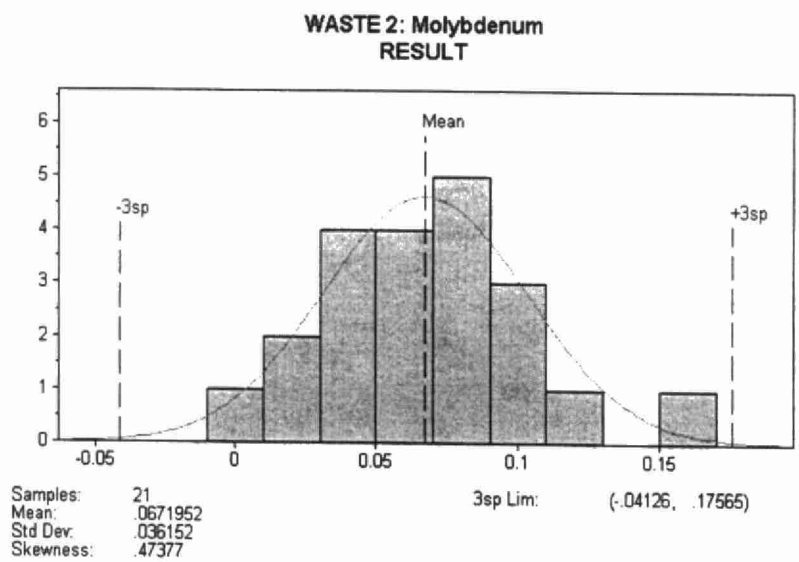
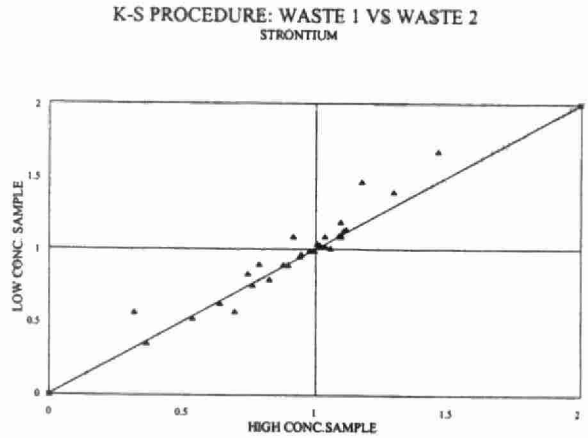
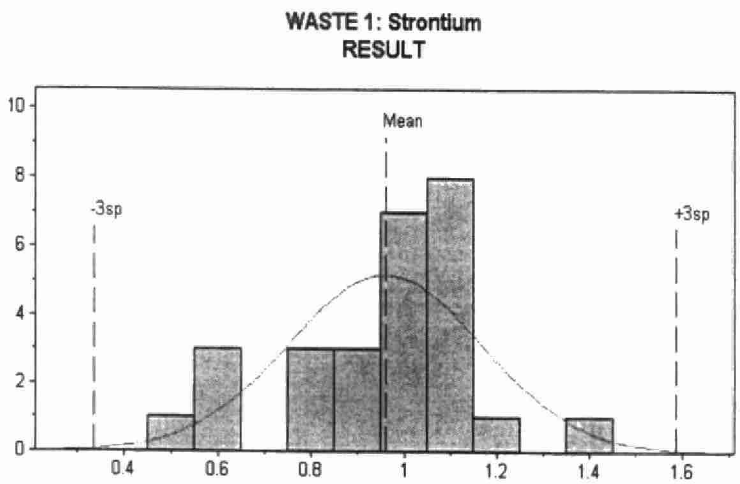


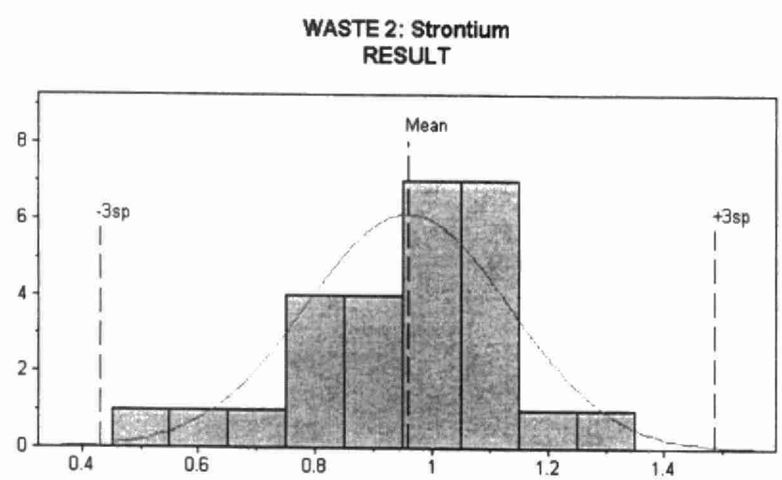
Figure A2-14: Strontium in Solid Wastes





Samples: 27
Mean: .96
Std Dev: .20839
Skewness: -.46028

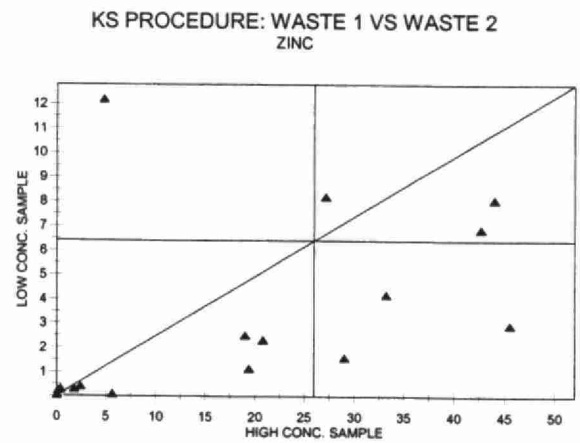
3sp Lim: (.33482, 1.5852)

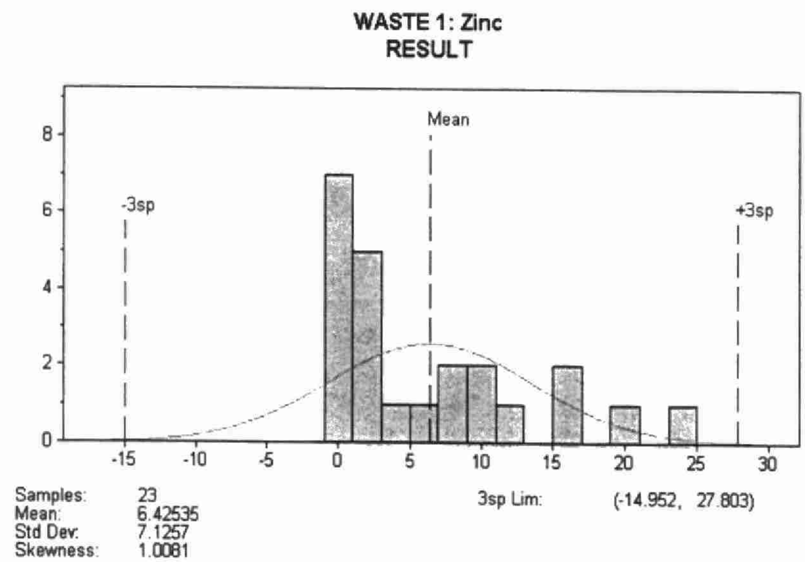


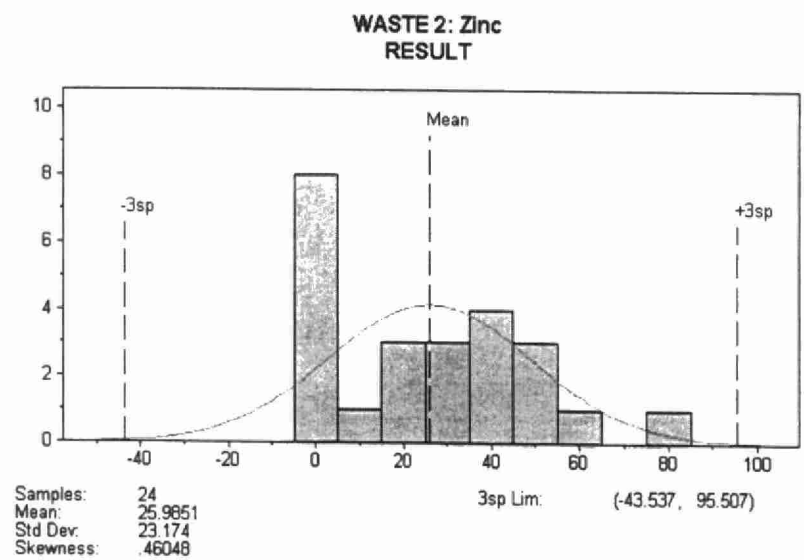
Samples: 27
Mean: .958408
Std Dev: .1757
Skewness: -.5292

3sp Lim: (.43132, 1.4855)

Figure A2-15: Zinc in Solid Wastes







APPENDIX 3 - METHODOLOGY SUMMARY

TABLE A3-1: WASTE EXTRACTION WEIGHTS, VOLUMES, FLUIDS & pH

	WASTE 1							WASTE 2						
ID Code	% Solids:	Initial pH	Final pH (after addition of acid & heating)	Leachate fluid selected	Sample Weight (g)	Volume of Leaching Fluid used (mL)	Final pH of Leachate	% Solids	Initial pH	Final pH (after addition of acid & heating)	Leachate fluid selected	Sample Weight (g)	Volume of Leaching Fluid used (mL)	Final pH of Leachate
2101	100	11.47	3.77	#1	40	800	10.8	100	11.41	0.94	#1	40	800	10.6
2102	96.88%	11.23	10.66	2	51.607	1000	8.60	96.98%	11.15	10.02	2	51.531	1000	8.00
2103	100	11.34	7.18	fluid # 2	50	1000	7.89	100	11.2	7.14	fluid # 2	50	1000	7.44
2104	97.0	10.69	6.29	2	50	1000	8.17	97.2	10.72	6.79	2	50	1000	7.63
2105	97.7	11.32	10.62	#2	51	1000	7.48	97.9	11.31	10.09	#2	51	1000	7.7
2106	100	11.43	10.22	#2	90	1800	7.76	100	11.43	9.80	#2	90	1800	7.47
2107	1.00	10.89	6.30	2	105.8	2112	7.2	1.00	10.93	6.72	2	104.4	2088	8.2
2108	97.5	>5.0	>5.0	2	100	1950	9.8	97.2	>5.0	>5.0	2	100	1944	9.6
2109		11	6.11	B	50	1000	5.35		10.85	6.1	B	50	1000	5.35
2110	100	10.88	10.54	2	101	2000	8.35	100	10.86	9.84	2	103	2000	8.77
2111	N/A	11.26	2.64	Solution A	10	200	10.81	N/A	11.22	3.32	Solution A	10	200	10.69
2112	100	11.1	7.6	2	50	1000	N/A	100	10.9	6.6	2	50	1000	N/A
2113	100%	11.41	7.38	#2 (pH 2.97)	25	500	7.39	100%	11.41	7.56	#2 (pH 2.97)	25	500	7.62
2116	100	12.84		Dilute Acetic Acid with a pH of 2.88	10	200	5.2	100	11.93		Dilute Acetic Acid with a pH of 2.88	10	200	5.2
2117	100	11.46	9.34	2	5	100	n/a	100	11.39	8.94	2	5	100	n/a
2118	100	10.37	10.32	2	100.01	2000	8.27	100	10.20	9.96	2	100.01	2000	7.75
2119	97.1	10.55	6.71	2	51.5	998.5	8.93	97.2	10.06	6.33	2	51.4	998.6	7.50
2120	98.60%	11.1	10.42	#2	20.293	405.9	6.05	98.30%	11.07	11.23	#2	20.158	402.3	6.86
2121	100	>5.0	>5.0	Fluid #2	40	800	7.2	100	>5.0	>5.0	Fluid #2	40	800	7.13
2122				TCLP extraction fluid #1	25.364	500	4.82					25.422	500	11.01

TABLE A3-1: WASTE EXTRACTION WEIGHTS, VOLUMES, FLUIDS & pH

	WASTE 1							WASTE 2						
ID Code	% Solids:	Initial pH	Final pH (after addition of acid & heating)	Leachate fluid selected	Sample Weight (g)	Volume of Leaching Fluid used (mL)	Final pH of Leachate	% Solids	Initial pH	Final pH (after addition of acid & heating)	Leachate fluid selected	Sample Weight (g)	Volume of Leaching Fluid used (mL)	Final pH of Leachate
2123	100	11.4	6.08	#2	25	500	6.88	100	11.3	6.42	#2	25	500	7.08
2124	100	11.10	9.59	Fluid # 2	50	1000	7.62	100	11.02	6.87	Fluid # 2	50	1000	7.20
2125	100	nil	8.82	# 2	20	400	8.03	100	nil	8.02	# 2	20	400	7.64
2126	100	11.3	10.6	#2	50	1000	7.3	100	11.5	9.7	#2	50	1000	7.4
2128	100	>5	>5	#2	50.594	1000	Not measured	100	>5	>5	#2	50.316	1000	Not measured
2130	98.2	10.94	7.03	#2	49.1	982	8.293	98.2	10.78	6.66	#2	49.1	982	7.472
2131	97	11.4	10.4	2	22.5	450	7.75	98	11.4	10.3	2	22.5	450	7.58
2132	96.9	11.1	6.32	2	51.6	1000	6.13	97.2	11.1	6.45	2	51.5	1000	6.38
2133	100	11.44	7.02	#2	25.071	500	7.91	100	11.32	6.42	#2	25.033	500	7.50

TABLE A3-2: WASTE EXTRACTION EQUIPMENT

ID CODE	Ext. Time	Filter Type	Filtration Equipment
2101	20 hrs	1 L Mason	Whatman 934-AH Glass Fibre Filter, used vacuum filtration
2102	20 hrs	dia=142mm, Glass Fibre pore size=0.7µ	KST 142 1.5L Pressure Filtration Device
2103	21 hrs	9.0cm Dia. 934-AH (Glass micro fibre filters)	Buchner funnel with suction
2104	17.5 hrs	14.2 cm grade ISI Ahlstrom glass, microfibre manually acid washed	Gelman Waste Pressure Filtration Unit, used vacuum filtration mode
2105	21 hrs	Whatman GF/F 4.25	Tap Aerator Vacuum Filter
2106	18 hrs	90mm GF/C	Vacuum Filtration
2107	16 hrs	142mm Millipore AP40, Glass Fibre Filter	Hazardous Waste Filtration Unit, Millipore
2108	18 hrs	0.7µm glass fibre filter (47mm)	Magnetic Millipore filter unit, 47mm
2109	18 hrs	47mm x .7U - GF	
2110	18 hrs	.7 glass fiber	Vacuum with 2L vacuum flask and magnetic filter funnel
2111	18 hrs	Glass fiber 1.1µm	Buchner Funnel & 500mL Vacuum flask
2112	18 hrs	Prefilter, glass, 0.5micron, 142mm	Filter holder, can exert pressure to 50psi
2113	18 hrs	Glass Fibre 142 mm	Millipore Pressure Filter
2116	18 hrs	0.45 micro cellulose acetate membrane filter paper	Vacuum pump, plastic filter device, 250mL filtering flask
2117	18 hrs	4.7 cm glass fibre, 0.7 µm pore size	Gelman filter apparatus, 250 mL capacity
2118	18 hrs	90mm, 0.70 µm TCLP filter	Vacuum pump, Gelmann 90 mm glass fritted stand and funnel
2119	18 hrs	0.7µm, 47mm diameter, glass fiber filter	Polyphenylsulfone, up to 500mL funnel capacity, vacuum operated, up to 8.9 cm diameter
2120	18 hrs	142mm, 0.7µm certified acid washed glass fibre filters	Stainless steel filtration unit 142mm diameter, minimum 1L capacity, capable of sustaining a pressure of 50psi, applied to the solution to be filtered
2121	18 hrs		Pressure Filtration Apparatus
2122	18 hrs	0.65pm Millipore white DAWP 142mm	Millipore Filtration Apparatus
2123	18 hrs	0.7µm, 47mm glass fibre	Nalgene polysulfone
2124	18 hrs	D. 142mm, 0.7µm, Borosilicate glass Fibre filter	Pressure Filtration Unit, D. 142mm, 1L capacity, 50psi
2125	18 hrs	0.45 µm-millipore HA	Vacuum filtration
2126	18 hrs	0.7 micron GF	Pressure filtration
2128	18 hrs	0.45 µm	Stainless steel filtration cup
2130	18 hrs	47mm 0.45 µm	vacuum
2131	18 hrs	GF/F 90mm Ø	Erlenmeyer flask, vacuum pump, filter
2132	18 hrs	0.45 µm/25mm	syringe filter
2133	19	#44 Whatman 3 µm retention	gravity

TABLE 3A-3: LEACHATE DIGESTION PROCEDURE

ID CODE	Digestion Fluid	Digestion Mode	Temp (°C)
2101	none	other	
2102	Aqua Regia	hot plate	95-100
2103	HNO_3	hot plate	65
2104	HNO_3	oven	103
2105	none	other	
2106	none	other	
2107	HNO_3	hot block	200
2108	HNO_3	hot block	
2109	HNO_3	hot block	
2110	HNO_3	hot plate	
2111	HNO_3	hot block	90
2112	none	other	
2113	HNO_3	microwave	95
2116	none	other	
2117	none	none	
2118	n/a	n/a	
2119	none	other	
2120	HNO_3	microwave	
2121	HNO_3/H_2O_2	hot block	95
2122	none	other	
2123	none	other	
2124	Aqua Regia	hot plate	95
2125	HNO_3	other	
2126	Aqua Regia	hot block	1 hour - 50; 1 hour - 95
2128	none	other	
2130	none	other	
2131	none	other	
2132	HNO_3	n/a	
2133	none	other	

TABLE A3-4: INSTRUMENTATION & CALIBRATION

ID CODE	Instrument	Calibration Mode	Matrix	Is Internal Standard (IS) Used	Results corrected for IS	Results corrected for Blanks	Dilution factor
2101	ICP-MS	multi pt	HNO3	yes	yes	no	10x
2102	ICP	Single pt	HNO3	no	no	no	Wastes: 1:10
2103	AAS	multi pt	HNO3			yes	Ca-1\2500, Mg- 1\250, Cr-1\10(Leaches)
2104	ICP	multi pt	HNO3	no		no	5x & Ca-100x
2105	ICP	Single pt	HNO3	no		no	
2106	ICP-MS	multi pt	HNO3				10x
2107	ICP	multi pt	HNO3	yes	yes	no	Wastes & Leach 2-10x
2108	ICP	multi pt	HNO3	no		yes	10x & 100x
2109	ICP-MS	multi pt	HNO3	yes	yes	no	100x
2110	ICP and ICP-MS	multi pt	HOAC			yes	10x
2111	ICP	multi pt	HNO3	no	no	yes	10x
2112	ICP	Single pt	HNO3	no		no	Wastes: 5x
2113	ICP	Single pt	HNO3	no	no	no	Wastes & Leaches: 5x
2116	ICP	Single pt	HNO3	no		no	Wastes: Ca-10x Leach 2: Cr, Na-10x
2117	ICP	multi pt: Al, Ca, Fe & Mg Single pt: all others	HNO3	yes; Y	yes	no	Wastes & Leach 2: 10x
2118	ICP	multi pt	HNO3	no	no	yes	10 x for Ca (Waste 1 & 2) 10 x for Zn (Waste 2)
2119	ICP	Single pt	HNO3	no		no	
2120	ICP-MS	multi pt	HNO3	yes	yes	yes	10x, 100x & 1000x
2121	ICP	multi pt	HNO3	no		no	Wastes: Ca-10x
2122	ICP	multi pt	HNO3	no	no	yes	
2123	ICP	Single pt	HNO3	no	no	yes	Wastes: 5x Leach 2: 2x
2124	ICP	Single pt	HNO3	no	no	no	Wastes & Leaches: 10x
2125	ICP-MS	multi pt	HNO3	yes	yes	yes	10x
2126	ICP	Single pt	1% HNO3	no	no	no	Wastes : 4x
2128	ICP	multi pt	HNO3	no	no	yes	Multiple (see comments)
2130	ICP	multi pt	HNO3	no		yes	Wastes: Ca -1000x, Mg-100x & Zn-10x
2131	ICP and ICP-MS	multi pt	HNO3	yes	yes	no	Standards: 10x
2132	ICP	Single pt	HNO3	no		yes	10x
2133	ICP and ICP-MS	Single pt	HOAC	yes	yes	yes	ICP: Wastes -10x

Legend

HNO3 - Nitric Acid

HOAC - Acetic Acid

Additional Comments

Laboratory 2123 received Leach 1 and Leach 2 partially frozen.

Laboratory 2128 noted that participation in this interlaboratory study was used for methodology development, as their ICP method is designed for drinking water analysis. Multiple dilutions were required for all of the samples.

APPENDIX 4 - LIST OF PARTICIPANTS

Accutest Laboratories Ltd., Nepean, Ontario

Activation Laboratories Limited, Ancaster, Ontario

ALS Environmental Vancouver, Vancouver, BC

AMEC Earth & Environmental Limited, Mississauga, Ontario

AMEC Earth & Environmental Limited, Edmonton, Alberta

Bodycote Materials Testing Canada Inc., Pointe Claire, Québec

Caduceon Environmental Laboratories, Ottawa, Ontario

Centre d'Expertise en Analyse Environnementale du Québec, Laval, Québec

Entech - A Div. of Agri-Service Laboratory Inc., Mississauga, Ontario

Enviro-Test Laboratories Manitoba Technology Centre Ltd., Winnipeg, Manitoba

Enviro-Test Laboratories, Calgary, Alberta

Enviro-Test Laboratories - Sentinel Division, Waterloo, Ontario

EnWin Laboratories & Water Research Centre, Windsor, Ontario

INCO LTD. Copper Cliff Central Lab, Copper Cliff, Ontario

Lakefield Research Limited, Lakefield, Ontario

Maxxam Analytics, Mississauga, Ontario

Maxxam Analytics Inc., Calgary, Alberta

Maxxam Analytique Inc. (Québec), Lachine, Québec

Norwest Labs, Surrey, BC

Norwest Labs, Edmonton, Alberta

Ontario Ministry of Environment, Laboratory Services Branch, PCLS, Etobicoke, Ontario

Paracel Laboratories Ltd., Ottawa, Ontario

Philip Analytical Services, Burlington, Ontario

Philip Analytical Services Inc., Mississauga, Ontario

PSC Analytical Services Inc., Burnaby, BC

PSC Analytical Services Inc., Anjou, Québec

PSC Analytical Services Inc., London, Ontario

PSC Analytical Services Inc., Edmonton, Alberta

Queen's Analytical Services Unit, Queen's University, Kingston, Ontario

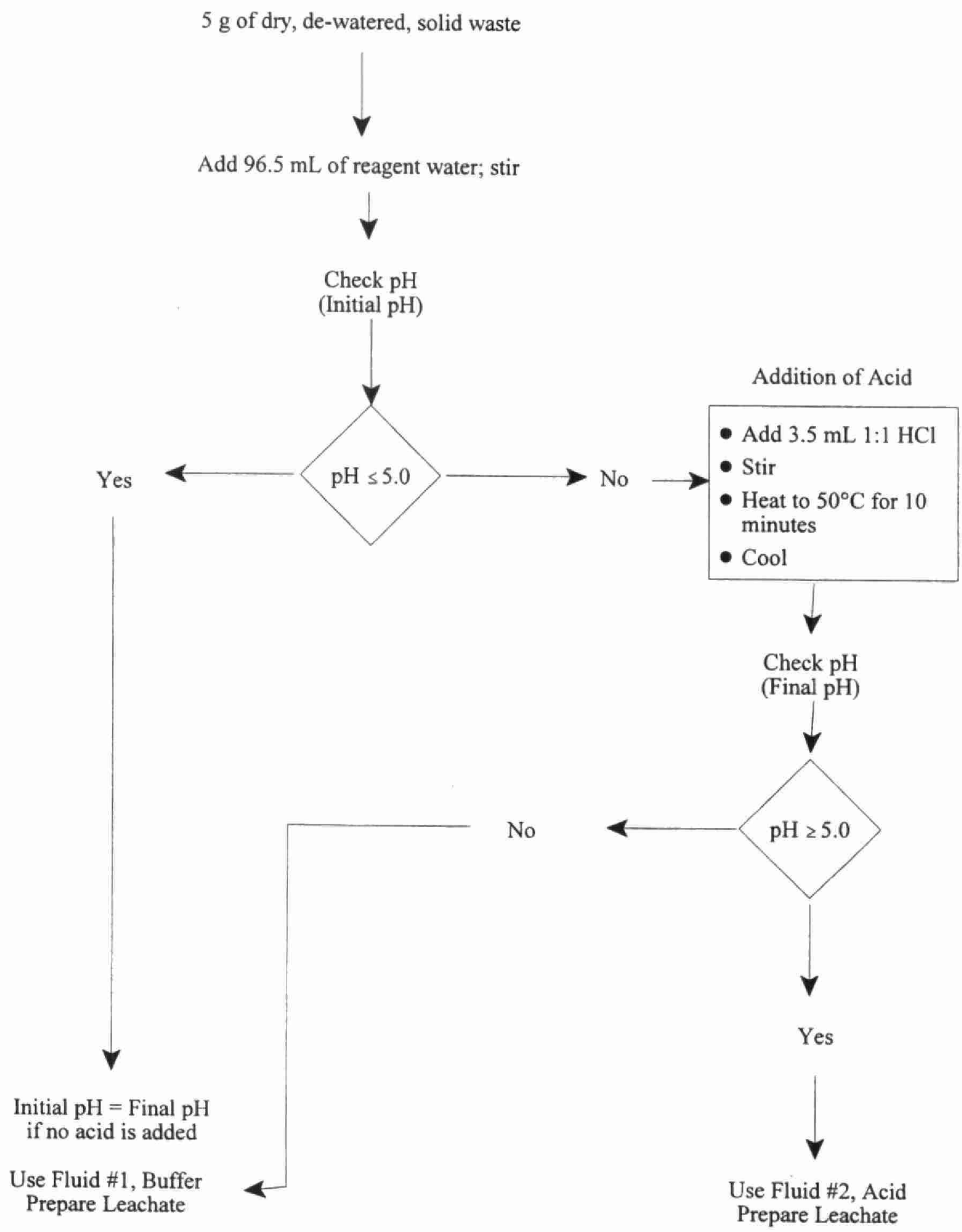
R & R Laboratories Ltd., Peterborough, Ontario

TESTMARK Laboratories Ltd., Sudbury, Ontario

York-Durham Regional Environmental Lab, Pickering, Ontario

APPENDIX 5 - FLOW CHART OF DETERMINATION OF TCLP EXTRACTION FLUID

DETERMINATION OF TCLP EXTRACTION FLUID



APPENDIX 6 - CORRESPONDENCE

Ontario

INVITATION FOR PARTICIPATION IN MOE INTERLABORATORY STUDY 02-1

The Laboratory Services Branch (LSB) of the Ontario Ministry of the Environment (MOE), is conducting an interlaboratory study in early 2002 to evaluate interlaboratory performance using the Toxicity Characteristic Leaching Procedure (TCLP), in support of Ontario Regulation 347, as ammended to O. Reg 558/00.

You are cordially invited to participate in this study.

The study will consist of two waste materials to be processed using either United States Environmental Protection Agency (US-EPA) Method 1311 or MOE-LSB method E9002, plus two solutions (leachate and/or standards) for analysis. The target analytes for all four samples are the following trace metals:

Table 1 - Target Analytes

Aluminum (Al)	Cobalt (Co)	Molybdenum (Mo)
Barium (Ba)	Copper (Cu)	Nickel (Ni)
Beryllium (Be)	Iron (Fe)	Strontium (Sr)
Cadmium (Cd)	Lead (Pb)	Vanadium (V)
Calcium (Ca)	Magnesium (Mg)	Zinc (Zn)
Chromium (Cr)	Manganese (Mn)	

The anticipated date for sample distribution is the week of February 25, 2002 and the results are expected to be reported by April 19, 2002.

If you are interested in participating, please complete the response form on the next page and return by December 31, 2001. Upon receipt of confirmation of participation, a copy of MOE-LSB method E9002 will be provided at no charge. If there are any questions you would like to ask before accepting, please do not hesitate to contact either of us by phone, fax or email.

Sylvia Cussion
phone:416-235-6348
Fax:416-235-6312
email:cussiosy@ene.gov.on.ca

Sathi Selliah
Phone:416-235-5700
Fax:416-235-6312
email:selliasa@ene.gov.on.ca

Mailing Address: Ministry of the Environment
Laboratory Services Branch
125 Resources Road
Toronto ON, M9P 3V6 Canada

MOE INTERLABORATORY STUDY 02-1

TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)

(PLEASE PRINT)

INSTITUTE/COMPANY:..

SHIPPING ADDRESS:...

....

....

....

....

TEL:...

FAX:...

Email:...

CONTACT NAME: ....

YES, We will participate in MOE ILS 02-1	
NO, We will not participate in MOE ILS 02-1	

SIGNATURE:...   DATE:.............................

Please return (or fax) this sheet to: Sylvia Cussion.
ILS Coordinator
Ministry of the Environment, Laboratory Services Branch
125 Resources Road
Toronto ON, M9P 3V6
Canada
Fax: 416-235-6312

Quality Management Unit

RE: MOE Interlaboratory Study 02-1

Thank you for your response to the invitation to participate in MOE Interlaboratory Study 02-1, Toxicity Characteristic Leaching Procedure (TCLP). Please find enclosed a copy of the Laboratory Services Branch (LSB) method E9002. Study participants are requested to use either the enclosed method **OR** US-EPA method 1311 for the processing of the solid samples provided as part of this interlaboratory study. Please note that the analytical methods listed in section 1.1.2 **ARE NOT** a requirement of this study. Participants may use their own method of choice to determine the target analytes in Table 1. A methodology questionnaire will be provided with the study samples.

Table 1 - Target Analytes		
Aluminum (Al)	Cobalt (Co)	Molybdenum (Mo)
Barium (Ba)	Copper (Cu)	Nickel (Ni)
Beryllium (Be)	Iron (Fe)	Strontium (Sr)
Cadmium (Cd)	Lead (Pb)	Vanadium (V)
Calcium (Ca)	Magnesium (Mg)	Zinc (Zn)
Chromium (Cr)	Manganese (Mn)	

Please do not hesitate to contact us if you have any questions regarding this interlaboratory study.

Sylvia Cussion
LSB ILS Coordinator
phone:416-235-6348
Fax:416-235-6312
email:cussiosy@ene.gov.on.ca

Sathi Selliah
LSB PE Coordinator
Phone:416-235-5700
Fax:416-235-6312
email:selliasa@ene.gov.on.ca

Quality Management Unit

February 26, 2002

RE: MOE Interlaboratory Study 02-1

Please find enclosed the samples and supporting materials for MOE Interlaboratory Study 02-1, Toxic Characteristic Leaching Procedure (TCLP). Please contact either of us immediately if any of the sample containers have been broken during shipping, and we will provide replacements.

The following has been provided to each participant:

2 Solid Waste Samples (Waste 1 and Waste 2)
2 Liquid Leachates (Leach 1 and Leach 2)
2 x 2 Ampouled Standards (2 each of 02-1-1 and 02-1-2)
An instruction sheet
Electronic report forms on 3.5" diskette: Data.xls and Questionnaire.xls

Please report all results and the methodology questionnaire ELECTRONICALLY by **April 19, 2002**.

Please do not hesitate to contact us if you have any questions regarding this interlaboratory study.

Your confidential Study ID Code is: **ID_Code**

Sylvia Cussion
LSB ILS Coordinator
phone:416-235-6348
Fax:416-235-6312
email:cussiosy@ene.gov.on.ca

Sathi Selliah
LSB PE Coordinator
Phone:416-235-5700
Fax:416-235-6312
email:selliasa@ene.gov.on.ca

INSTRUCTION SHEET

MOE INTERLABORATORY STUDY 02-1
TOXIC CHARACTERISTIC LEACHING PROCEDURE (TCLP)

SOLID WASTES: WASTE 1 and WASTE 2

Each sample consists of 110 g of a solid waste. Both samples are to be processed using either MOE-LSB Method E9002 (provided in a previous mailing) OR US-EPA Method 1311. The resulting leachates should be analyzed for the Target Analytes (Table 1) using the participant's routine analytical methods. Please record all relevant information regarding the preparation and analysis of these samples in the Excel spreadsheet Questionnaire.xls. All analytical data are to be reported in the Excel spreadsheet Data.xls.

LIQUID LEACHATES (LEACH 1 and LEACH 2)

Each sample consists of approximately 250 mL of leachate that has been prepared according to MOE-LSB Method E9002. Both samples should be analyzed for the Target Analytes (Table 1) using the participant's routine analytical methods. Please record all relevant information regarding the preparation and analysis of these samples in the Excel spreadsheet Questionnaire.xls. All analytical data are to be reported in the Excel spreadsheet Data.xls.

AMPOULED STANDARDS (02-1-1 and 02-1-2)

Each ampoule contains approximately 20 mL of the Target Analytes standard at different concentrations. They have been preserved with 2% Nitric Acid. Both solutions are for direct instrumental analysis (no pretreatment required) and should be analyzed for the Target Analytes (Table 1) using the participant's instrumental method used for the Waste and Leachate samples. Please record all relevant information regarding the analysis of these standards in the Excel spreadsheet Questionnaire.xls. All analytical data are to be reported in the Excel spreadsheet Data.xls.

TARGET TRACE METALS

Table 1 - Target Analytes		
Aluminum (Al)	Cobalt (Co)	Molybdenum (Mo)
Barium (Ba)	Copper (Cu)	Nickel (Ni)
Beryllium (Be)	Iron (Fe)	Strontium (Sr)
Cadmium (Cd)	Lead (Pb)	Vanadium (V)
Calcium (Ca)	Magnesium (Mg)	Zinc (Zn)
Chromium (Cr)	Manganese (Mn)	

DATA REPORTING

All analytical data are to be reported electronically using the enclosed Excel spreadsheet Data.xls. All relevant information regarding the preparation and analysis of the samples is to be recorded in the Excel spreadsheet Questionnaire.xls and also reported electronically. All result are due by **April 19, 2002** and should be reported via e-mail to either Sylvia Cussion at cussiosy@ene.gov.on.ca or Sathi Selliah at selliasa@ene.gov.on.ca, or by returning the diskette with the completed report forms.

MOE ILS 02-1
RESULTS REPORTING SHEET
ID CODE:

Analyte	INSTRUMENT	INSTRUMENT STANDARDS		LEACHATES		SOLID WASTE	
	TECHNIQUE	02-1-1	02-1-2	LEACH 1	LEACH 2	WASTE 1	WASTE 2
	(eg AAS, ICP)	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
Aluminum							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Molybdenum							
Nickel							
Strontium							
Titanium							
Vanadium							
Zinc							

MOE INTERLABORATORY STUDY 02-1
TOXIC CHARACTERISTIC LEACHATE PROCEDURE (TCLP) METHODOLOGY QUESTIONNAIRE
LAB ID CODE:

Date Samples Received:

SAMPLE PREPARATION

Date Analysis Initiated:

	WASTE 1	WASTE 2
% Solids:		
Initial pH:		
Final pH (after addition of acid & heating):		
Leachate fluid selected:		

LEACHATE PROCEDURE

	WASTE 1	WASTE 2
Date Analysis Initiated:		
Sample Weight:		
Volume of Leaching Fluid used:		
Extraction Time:		
Final pH of Leachate:		
Size & Type of Filter:		
Describe Filtration Equipment:		
pH of Final Preserved Sample (leachate):		

SAMPLE ANALYSIS

Date Analysis Initiated:

	WASTE 1	WASTE 2	LEACH 1	LEACH 2
Digestion Acid (please select below):				
Aqua Regia (1:3 HNO3:HCl)				
HNO3 only				
Other (please specify)				
No digestion performed				
Digestion Procedure (please select below):				
Hot Plate				
Hot Block				

MOE INTERLABORATORY STUDY 02-1
TOXIC CHARACTERISTIC LEACHATE PROCEDURE (TCLP)
METHODOLOGY QUESTIONNAIRE
LAB ID CODE:

	WASTE 1	WASTE 2	LEACH 1	LEACH 2	02-1-1	02-1-2
Microwave						
Other (please describe)						
Temperature (°C)						
Date of Instrumental Analysis:						
Instrumental Technique (please select below):						
AAS						
GFAAS						
ICP						
ICP-MS						
Other (please describe)						
What type of calibration was used:						
Single point						
Multi-point						
What is the matrix of your calibration standards?						
H2O						
HNO3						
Acetic Acid						
Leachate A						
Leachate B						
Did you use an Internal Standard?						
If YES, please describe						
Did you correct the results with respect to the Internal Standard?						
Did you Blank Correct?						
Was sample dilution required?						
If YES, please provide the dilution factor.						